THE NICKEL BULLETIN

A MONTHLY SUMMARY OF CURRENT INFORMATION

JULY/AUGUST, 1957

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ABSTRACTS OF CURRENT PUBLISHED INFORMATION ON NICKEL AND ITS ALLOYS

GENERAL

Creep-Rupture Testing of Metals and Alloys

M. J. MANJOINE: 'Combination Creep-Rupture Test Specimen.'

A.S.T.M.Bull.,1 957, No. 222, May, pp. 36–7; *T.P.* 104.

In the development of new alloys or in improving heat-treatment schedules for established materials, as well as in the acceptance testing of materials for high-temperature service, it is usually necessary to determine both creep-rupture strength and notch sensitivity. Such information is secured by creep-rupture tests on smooth and on (separate) notched specimens and, since notch-sensitivity may vary for material in different positions in any one batch, the notched test section must be taken as close as possible to the place from which the smooth-bar test section is taken.

This paper describes (with drawing) apparatus in which tests on smooth and notched sections can be combined on one specimen, thus saving time and ensuring a close degree of uniformity in the material tested. The usefulness of the method is discussed as exemplified by tests on an austenitic steel for high-temperature service (Discaloy) and in relation to its use for development of high-temperature alloys in general, and heat-treatments for such materials.

High-Temperature Hardness Testing

See abstract on p. 139.

Influence of Surface Films on Friction and Wear

E. E. BISSON, R. L. JOHNSON, M. A. SWIKERT and D. GODFREY: 'Friction, Wear and Surface Damage of Metals as Affected by Solid Surface Films.'

Nat. Advisory Committee for Aeronautics, Report 1254, 1956; 19 pp.

The report is based on research data obtained during a series of investigations carried out from 1946 to 1954, on lubrication, friction and wear. As is well known, the presence of a contaminating film* between sliding surfaces can have a marked effect on friction, wear and surface damage: some contaminants are beneficial (lubricants), others are harmful (abrasives).

In the experimental work reported, factors known to exert a significant influence on the effect of surface films variously formed were individually studied, to clarify their influence on the service behaviour of sliding metals.

The tests applied provided for conditions of kinetic friction, static friction and reciprocating sliding: a general description is given of the essential features of each form of test. In most of the studies both of the contacting specimens were of steel and the films under investigation were applied to the disc member of the unit. Some tests were made also with nickel- or copper-base alloys.

The results of the various series of experiments, which are reported in considerable detail, demonstrate that, as predicted by friction theory, wear and surface damage on metals can be reduced by solid surface films. The ability of materials to form surface films which prevent welding is a major factor in determining the wear of dry and boundary-lubricated surfaces. Films of graphitic carbon on cast irons, of NiO on nickel alloys and of FeO and Fe_3O_4 on ferrous materials were found to be beneficial. Abrasive films such as Fe_2O_3 or MoO_3 were definitely detrimental. The evidence obtained in these tests suggests that the importance of oxide films to friction and wear processes has not until now been fully appreciated.

The chemical-reaction films FeS and $FeCl_2$ were both effective in the prevention of surface welding: because of its lower shear strength, $FeCl_2$ was the more effective in reducing friction.

Solid lubricants such as molybdenum disulphide and graphite are considered to warrant careful study for use under severe conditions, e.g., at high temperatures and high sliding velocities. Both these lubricants can be bonded to many surfaces by the use of a resin-forming liquid. Rolling-contact bearing experiments showed that effective lubrication could be maintained at temperatures of 1000°F. (540°C.) by injection of minute amounts of these solids. In the presence of oxygen MoS_2 oxidizes to MoO_3: the reaction occurs at a very slow rate at 750°F. (400°C.), but becomes more rapid at temperatures above 1050°F. (565°C.).

The report is accompanied by an extensive selected *bibliography* on the theoretical and practical aspects of friction and wear.

Etching by Gas-Ion Bombardment

J. B. NEWKIRK and W. G. MARTIN: 'A Method for the Etching of Metals by Gas-Ion Bombardment.'

Trans. Amer. Soc. Metals, 1957, vol. 50, *Preprint* No. 8; 6 pp.+ tables and figures.

The technique described in this paper is an improved modification of that proposed by Padden and Cain (*Metal Progress*, 1954, vol. 66, July, pp. 108–12). In the new procedure krypton gas is used, and a magnetic field is applied to increase the efficiency

* A contaminant is defined as any material other than those comprising the sliding surfaces.

of ionization. The method is in routine use in the laboratories of the General Electric Company, Schenectady.

The paper includes full details of apparatus and technique by which it is possible to etch metallographic specimens more rapidly, and with less heating of the specimen than previously occurred.

Use of Electrochemical Methods in Metallographic Examination of Steels

See abstract on p. 146.

Metal Powders: Production Methods, etc.

'Metal and Alloy Powders: A Directory.'
Materials and Methods, 1957, vol. 45, Apr., pp. 163, 165, 167 and 169.

A tabular summary of the powder metals now commercially available, the methods by which they are made, and the mesh sizes and degrees of purity in which they are produced. This information is supplemented by the names of powder-metal suppliers in the U.S.A.

NICKEL

A Quarter-Century's Progress in the Nickel Industry

L. B. PFEIL: 'Some Aspects of Progress in the Nickel Industry.'
Jnl. Inst. Metals, 1956–57, vol. 85, July, pp. 457–61.
Reprint of Presidential Address to the Institute of Metals, delivered April 30th, 1957.

The review is intentionally highly selective. Particular emphasis is laid on progress which has been made, during the past twenty-five years, in methods used for prospecting (e.g., the large-scale use of airborne magnetometric surveying), and on improvements which have been introduced in mining, extraction and refining methods. Some of the outstanding features of methods used by The International Nickel Company and The Mond Nickel Company are considered. The closing section of the Address contains reference to some of the major applications of nickel, as a constituent of stainless steels and of constructional steels, in plating, and in the field of high-temperature engineering. Attention is directed to the significance of nickel-base alloys for aircraft jet engines and gas turbines, and, in connexion with that development, mention is made of the important influence of trace constituents, on the properties obtainable in high-temperature alloys and other materials.

Nickel-Copper Smelters and Nickel Refineries in Canada

See abstract on p. 114.

Dynamic Elastic Modulus of Nickel at Elevated Temperatures

See abstract on p. 128.

High-Temperature Tensile Properties of Nickel, Copper-Nickel Alloys and Copper

See abstract on p. 115.

Influence of Ceramic Coatings on Creep Properties

See abstract on p. 132.

Nickel and Nickel-Clad Materials for Electron Tubes

AMER. SOC. TESTING MATERIALS: 'Report of Committee F-1 on Materials for Electron Tubes and Semi-Conductor Devices.'
A.S.T.M. Preprint 61, June 1957; 18 pp.

The Committee recommends for publication as *Tentative* the following specifications:

(*a*) 'Clear Nickel-Clad and Nickel-Plated Steel Strip for Electron Tubes', pp. 5–6.

The specification lays down requirements for the chemical composition of the carbon-steel base metal, the nickel cladding and plating, the method of bonding, two thicknesses each of cladding and plating (with provision for methods of inspection testing), temper of strip, finish, dimensions and tolerances, coiling or spooling, packing and marking. For the cladding material the following percentage limits are specified: nickel+cobalt 99·00 min., carbon 0·15 max., copper 0·15 max., iron 0·20 max., magnesium 0·15 max. (preferably 0·10 max.), manganese 0·30 max., silicon 0·10 max., sulphur 0·008 max., titanium 0·005 max. The nickel plating is required to be of nickel+cobalt 99·95 min. per cent. purity.

(*b*) 'Aluminium-Clad Steel Strip and Nickel-Steel-Aluminium Composite Strip for Electron Tubes', pp. 7–9.

The specification covers steel strip clad on both sides with aluminium and steel strip clad with aluminium on one side and with nickel on the other. The composition limits for the base carbon steel and for the nickel of the cladding are as laid down in the previous specification. Limits for iron, silicon and zinc are specified for the aluminium used in cladding. Other clauses of the schedule are also closely similar to that governing nickel-clad steel, with the addition of a section dealing with metallographic procedure for examining cladding thickness and detecting interface constituents in aluminium-clad strip.

(*c*) 'Clear Nickel Strip for Electron Tubes', pp. 10–11.

It is specified that the nickel strip used for cathodes shall conform to A.S.T.M. Designation F-239 (see *Book of A.S.T.M. Standards*, 1955, Part 6). For nickel which is to be used for parts other than cathodes the limits are as shown for nickel employed for cladding, *see above*. The specification also covers temper, finish, tolerances, etc.

(*d*) 'Carbonized Nickel Strip and Carbonized Nickel-Plated and Nickel-Clad Steel Strip for Electron Tubes', pp. 12–13.

The nickel base to be carbonized is required to conform to the compositional limits laid down in the specification for Clear Nickel-Clad and Nickel-Plated Steel Strip referred to in item (*a*) p. 108, or to the F-239 specification referred to in item (*c*). Temper, finish, dimensions and tolerances, etc., are in the usual forms, and details are given of methods of testing for carbon-loss by firing of carbonized materials, to ensure an acceptable standard of quality for operation in electron-tube applications.

(*e*) 'Nickel-Alloy Cathode Sleeves for Electronic Devices', pp. 14–18.

The specification covers 'cathode sleeves, seamless, welded and drawn, or lockseam, for use as indirectly heated cathodes in electronic devices'. The compositional limits laid down are shown in the table below, and it is specified that orders for cathode sleeves furnished to this specification shall include information relating to method of fabrication, alloy grade (see below), diameter, wall thickness, length, bead diameter, bead location, and number of cathode sleeves required.

The specification includes requirements relating to sampling for analysis, inspection tests, the physical characteristics of the products, etc.

Corrosion-Resisting Applications of Monel and Nickel

See abstract on p. 143.

Sulphur-containing Nickel Catalysts

H. PINES and W. S. POSTL: 'Modified Nickel Catalyst for the Hydrogenolysis and Alkylation of Aromatic Hydrocarbons.'

Jnl. Amer. Chemical Soc., 1957, vol. 79, Apr. 5, pp. 1769–70.

Note reporting that when small amounts of sulphur-containing compounds are added to a nickel hydrogenolysis catalyst the latter is 'modified' in such a way as to render it capable of catalyzing reactions which have previously been associated with strong acid catalysts. Some of the following reactions have been observed to occur in the presence of nickel catalysts so modified: dealkylation, transalkylation, alkylation, and internal alkylation of benzenoid hydrocarbons. Some typical reactions carried out with the modified catalysts are described.

Nickel Catalysts: Influence of Particle Size

R. F. BURKE, R. L. MOTARD, L. N. CANJAR and R. B. BECKMANN: 'The Effect of Catalyst Granule Size on the Hydrogenation of Benzene over a Supported Nickel Catalyst.'

Jnl. Applied Chemistry, 1957, vol. 7, Mar., pp. 105–9.

In connexion with the study of the hydrogenation of benzene to *cyclo*hexane over nickel and nickel

Chemical Composition Limits for Cathode Sleeves or Tubing

0·005-in. Thick and Less*

Grade	Cu max %	Fe max. %	Mn %	C max. %	Mg %	Si %	S max. %	Ti %	W %	Ni+Co min. %
Active Alloy Type(a)										
3	0·20	0·20	0·20 max.	0·08	not specfd.	0·15-0·25	0·008	not specfd.	—	99·00
4	0·04	0·10	0·02-0·10	0·08	0·01 max.	0·15-0·25	0·005	0·02 max.	—	99·25
6	0·04	0·05	0·02 max.	0·08	0·01 max.	0·15-0·25	0·005	0·02 max.	—	99·25
7	0·20	0·20	0·20 max.	0·10	0·01-0·10	0·02-0·06	0·008	0·02 max.	3·50-4·50	94·50
10	0·15	0·20	0·30 max.	0·10	not specfd.	0·10 max.	0·008	not specfd.	—	99·00
11	0·20	0·20	0·20 max.	0·08	0·01-0·10	0·01-0·05	0·008	not specfd.	—	99·10
Passive Alloy Type(a)										
21	0·04	0·05	0·02 max.	0·05(*b*)	0·01 max.	0·01 max.	0·005	0·01 max.	—	99·50
22	0·04	0·05	0·02 max.	0·05(*b*)	0·01 max.	0·02 max.	0·005	0·01 max.	—	99·50

* A further table gives typical chemical compositions of cathode nickel alloys currently furnished to the specifications for the respective grades.

(*a*) Electron-emission activity of an oxide-coated cathode base material is its relative ability to provide reducing elements to the oxide coating: it is measured by the rate at which, and the degree to which, cathode electron emission develops under controlled conditions.

(*b*) Generally the carbon content of these alloys in the form of lockseam sleeves averages 0·01 per cent. for each 0·001 in. of thickness; seamless sleeves average slightly higher.

oxide supported on silica gel (*ibid.*, 1957, vol. 7, p. 1), an examination was made of the effect of granule size of the catalyst. The tests were carried out at 225°, 250° and 275°C.

It was found that with increasing state of subdivision, down to a certain granule size, the experimental velocity constant, k_e, is proportional to the external surface area of the catalyst. Further subdivision was found to have no effect on activity, since, when the critical granule size is reached, a sufficient area is exposed to permit of all the catalyst being adsorbed on the surface of the carrier.

Nickel-Tungsten Catalysts in Hydrogenation of Naphthas

'Hydrofining Thermally Cracked Naphthas.'

Industrial and Engineering Chemistry, 1957, vol. 49, Apr., p. 656.

Conjugated diolefins and sulphur compounds usually impair the stability and other performance characteristics of cracked naphthas. They appear in low concentrations, and are difficult to remove without also removing desirable substances such as high-octane monoolefins. The petroleum industry has devoted much research towards the solution of the problem, by application of mild hydrogenation for removal of the undesirable constituents, without undue saturation of olefins. This note is a contribution to the subject. A nickel-tungsten catalyst was employed in the experiments described.

Nickel Catalyst in Hydrocarbon Re-Forming Process

'This Re-Forming Process is Different.'

Chemical Engineering, 1957, vol. 64, Apr., pp. 152, 154.

Notes on the CAR (catalytic autothermic re-forming) process, developed in Europe and recently introduced into the U.S.A. Continuous autothermic catalysis is used to produce a hydrogen-rich fuel gas or ammonia synthesis gas. The catalyst used in the process is an impregnated material containing free nickel, specially developed to give high activity, to work at high temperature, and to ensure long life.

Self-Discharge in Nickel-Alkaline Batteries

A. L. PITMAN and G. W. WORK: 'Alkaline Storage Batteries. The Self-Discharge of the Positively Charged Nickel-Oxide Electrode.'

Part I. The Rôle of Water in the Process.

Part II. Potential as a Function of Time on Open Circuit and as a Function of the Active Oxygen on the Nickel-Oxide Plates.'

U.S. Naval Research Laby. Reports 4844 and 4845, Oct., 1956.

Part I. Alkaline nickel-oxide/iron storage batteries were charged and put on open circuit. The conclusion is that since the rate of oxygen evolution on open circuit is proportional to the square root of the thermodynamic activity of water in the electrolyte, the oxygen deposited on the nickel sesquioxide requires interaction with water for its evolution.

Part II. The potential on decay changes linearly with the quantity of oxygen on the electrode. The authors advance the hypothesis that there is adsorption of oxygen on, and desorption from, the Ni_2O_3.

Nickel Overlay on Paper-Press Rolls

'How Brunswick Repairs Grooved Press Rolls.'

Paper Trade Jnl., 1957, vol. 141, May 13, pp. 46-7.

Report of service experience in the mills of Brunswick Pulp and Paper Company, where the use of a nickel overlay, applied by the Sigma welding process, on grooved cast-iron rolls has reduced to a minimum time lost in maintenance, while simultaneously ensuring a clean and unstained product. As an example of the savings effected, it is reported that one set of rolls surfaced with nickel has been in operation for two years and another set for a year. Both are still in excellent condition. This service compares with a maximum of six months' life for rolls which were not so treated. The use of the Sigma welding process has resulted in a marked saving in time and cost of the surfacing operation, by comparison with other methods of overlaying.

Electrodes for Arc Welding of Monel and Nickel

See abstract on p. 119.

Spectrochemical Analysis of Nickel Alloys: Specifications

See abstract on p. 120.

Determination of Nickel in Aluminium Alloys

See abstract on p. 121.

Heat of Combustion of Nickel Carbonyl

A. K. FISCHER, F. A. COTTON and G. WILKINSON: 'Heat of Combustion and Formation of Metal Carbonyls. II. Nickel Carbonyl.'

Jnl. Amer. Chemical Soc., 1957, vol. 79, May 5, pp. 2044-6.

The heat of combustion of liquid nickel carbonyl, $Ni(CO)_4$, has been determined as $\Delta H^\circ_{298} = -282{\cdot}2 \pm 0{\cdot}5$ kcal./mole. For the reaction $Ni(s) + 4CO(g, 1\ atm.) = Ni(CO)_4(g, 1\ atm.)$ a value of $-39{\cdot}1 \pm 0{\cdot}5$ kcal./mole is calculated for ΔH°_{298}. The authors discuss the relation of these values to the various highly divergent results previously reported in the literature.

New Superstructure of Nickel Oxide

G. I. FINCH and K. P. SINHA: 'A New Superstructure of Nickel Oxide.'

Trans. Faraday Soc., 1957, vol. 53, May, pp. 623-7.

The work reported demonstrates that, under certain conditions, a new superstructure of nickel oxide can be stabilized in thin films. Electron-diffraction

examination indicates that the substance is a γ nickel oxide which is a metal-excess spinel of the composition $Ni_{28}O_{32}$.

ELECTRODEPOSITION AND OTHER COATING METHODS

Microthrowing Power: Review

E. B. LEFFLER and H. LEIDHEISER: 'Microthrowing Power: A Literature Search.'

Plating, 1957, vol. 44, Apr., pp. 388–96.

The necessity of developing techniques for electroplating in the pores of powder compacts, for retaining high fidelity in gramophone masters, and for depositing within recesses of intricately shaped parts, has helped to build up a fund of know-how among electrodepositors, but most of this knowledge is still retained as part of the art of electroplating, and but little has been written on the subject. The purpose of this report is 'to summarize published experimental and theoretical work concerned with the electrodeposition of metals in isolated surface cavities'. The review is intended to serve as a background for planning the experimental work which is to be undertaken by Research Project No. 17 of the American Electroplaters' Society.

The report is divided into four sections. The first is a summary discussion of the significance of the terms levelling, throwing power, covering power, etc.; the second section reviews pertinent experimental work on micro-throwing power and levelling: the third section gives a survey of the limited theoretical work which has been concerned with deposition within isolated recesses and cavities. The fourth section outlines the experimental work which will be undertaken in the near future by the Research Committee.

Estimation of Throwing Power of Plating Solutions

R. V. JELINEK and H. F. DAVID: 'Throwing Index: A New Graphical Method for Expressing Results of Throwing-Power Measurements.'

Jnl. Electrochemical Soc., 1957, vol. 104, May, pp. 279–81.

The authors propose a new direct method for expressing the results of throwing-power measurements made on plating solutions in a conventional rectangular throwing-power box with plane parallel electrodes. The metal-distribution ratio (M) is plotted *vs.* the linear ratio (P), on arithmetic co-ordinates. The reciprocal of the slope of this plot, designated the 'throwing index', represents a direct measure of the throwing power of the solution. Experimental data on some nickel solutions and other electrolytes are recorded to demonstrate the advantages of the method.

A Quarter-Century's Progress in the Nickel Industry

See abstract on p. 108.

Fuchsin as Brightener in Nickel Deposits

J. L. DYE and O. J. KLINGENMAIER: 'Inclusion of Fuchsin in Bright Nickel Deposits.'

Jnl. Electrochemical Soc., 1957, vol. 104, May, pp. 275–9.

This investigation was carried out to determine whether an organic brightener of the second class, as typified by fuchsin, is included in nickel electrodeposits without complete decomposition. Having demonstrated by solution of the nickel in hydrochloric acid that some fuchsin was recovered, a study was made of the effect of brightener concentration and of the current density at which the solution was operated. The amount of fuchsin recovered per gram of nickel was roughly linear in fuchsin concentration and it decreased with increase of the current density employed in deposition.

Plating Tests on Nickel Anodes

H. OFFERMANNS, H. VON FELD, S. KALBITZER and W. SCHRÖDER: 'The Use of Cast, Rolled and Electrolytic Nickel Anodes in Various Types of Nickel-Plating Solution.'

Metalloberfläche, 1956, vol. 10, Jan., pp. 22–3.

Very conflicting views are held amongst platers with regard to the relative usefulness of various types of nickel anode. The work described in this paper was carried out to obtain additional information on this subject, by studying the behaviour of various anodes in matt nickel-plating solutions and in two commercial bright nickel-plating baths.

The conditions used were as closely parallel as possible to those generally obtaining in plating shops. Particular attention was directed to the following aspects:

(1) The purity of the anodes.

(2) Solubility of the respective types of anode (anodic current efficiency).

(3) Formation of sludge.

(1) *Purity*

Examination of specimens of the three types of anode showed considerable differences in degree of purity. Typical figures are given below:

(*a*) Cast anodes—copper 0·1, iron 0·51, manganese 0·14, per cent.

(*b*) Rolled anodes—copper 0·05, iron 0·16, manganese 0·15, per cent.

(*c*) Electrolytic anodes—copper traces, iron 0·04 per cent., manganese traces.

(2) *Solubility*

There is a general impression that cast anodes dissolve particularly well, that rolled anodes are not so good, and that electrolytic anodes behave relatively badly, but the tests reported in this paper give no confirmation of this view.

(3) *Sludge Formation*

All types of anode examined gave rise to some form of sludging, but the type and quantity of the sludge varied. Whereas the electrolytic anodes gave off only coarse nickel platelets (which by reason of their

high specific weight fell quickly to the bottom of the tank), the rolled anodes, and still more the cast type, were characterized by formation of a light-brown-to-black sludge.

Corrosion-Resistance of Electrodeposited Nickel/Chromium Coatings on Steel

C. H. SAMPLE: 'The Corrosion Behaviour and Protective Value of Copper/Nickel/Chromium and Nickel/Chromium Coatings on Steel.'

Reprint of paper abstracted in *Nickel Bulletin*, 1957, vol. 30, No. 6, p. 86.

Issued by INTERNATIONAL NICKEL CO., INC.

Surface Treatments for Use on Low-Alloy Steels

S. TOUR: 'Surface Treatments for Low-Alloy Steels.' *Wright Air Development Center, Tech. Report* 54-209, Nov., 1954 (recently declassified); 203 pp. *P.B.* 121,507.

This report has been prepared as a reference document, to be used as a guide to U.S. Air Force contractors in the selection of surface treatments for the improvement of corrosion- and oxidation-resistance, with a minimum use of 'critical' materials. An evaluation is made of 40 metallic and 9 paint-type coatings applicable to carbon and low-alloy steels. The effect of temperature on the respective coatings, and their resistance to corrosion and abrasion is considered, also the influence of the various types on the properties of the base metal, and the formability and weldability of the coated steels.

A brief description is given of the processes currently used for applying the various forms of coating, and reference is directed to some of the further information available in the literature. The report is amplified by a glossary giving definitions of many of the terms used in the text.

The scope of the reference material included in the report is indicated below:—

Aluminium Coatings (Aluminizing, cladding, hot dipping, Calorizing, vacuum evaporation and electrodeposition).

Nickel and Nickel-Alloy Coatings (Electrodeposited nickel and nickel-phosphorus types, electroless nickel, Corronizing, coatings deposited by decomposition of the carbonyl, nickel and nickel-alloy cladding).

Diffusion Coatings (Chromizing, electrodeposition of chromium, siliconizing).

Miscellaneous Metallic Coatings (Cadmium, chromium, copper, silver, tungsten-alloy, zinc, and molybdenum coatings: Peen plating; stainless-clad steel).

Paint Coatings of various types.

Nickel Plating of Aluminium Bronze

I. W. MARKOVITCH: 'Plating on Aluminium Bronze.' *Plating*, 1957, vol. 44, Apr., p. 387.

After unsuccessful trials with various pre-plating treatments, the following cycle has been evolved for the treatment of aluminium-bronze forgings which are to be plated. It is found to ensure satisfactory adhesion of the deposited metals.

(*a*) Electro-clean in a brass cleaner.

(*b*) Dip in hydrochloric acid (15 per cent. by vol.; room temperature).

(*c*) Dip in a nitric-acid/ammonium-bifluoride solution of the following composition:

Nitric acid (conc.)	.. ..	100 fl. oz.
Ammonium bifluoride	.. ..	11 „
Water	.. ..	.. to make one gallon.

Operated at room temperature: immersion for 3 seconds.

(*d*) Repeat operations (*a*) and (*b*).

(*e*) Strike in a copper-cyanide solution.

(*f*) Plate with nickel.

Rinse thoroughly between the respective steps of the cycle.

The nitric-acid/ammonium-bifluoride solution removes the aluminium and silicon from the surface of the alloy, etching it very slightly, and thus providing a good adhesion base for the copper strike and the nickel plating. It is considered that the same procedure should give good results in preparation for the deposition of other metals.

Electroplating on Zirconium

W. C. SCHICKNER, J. G. BEACH and C. L. FAUST: 'Electroplating on Zirconium using Replacement Indium Deposits.'

U.S. Atomic Energy Commission, Report BMI-809, Feb. 2, 1953 (recently declassified): 15 pp.

Previous reports in this series (describing research at the Battelle Memorial Institute) dealt with alloy-bonded nickel and iron on zirconium, and with improvements in the alloy-bonding process and its application to zirconium-tin alloys.

In this third report it is shown that electroplated nickel on zirconium has a bond of 18,000 p.s.i. modulus of rupture when plated over an indium chemical-replacement coating produced by treatment in a fluoborate solution. This value in modulus of rupture represents the best as-plated bond yet obtained on zirconium.*

The process is simple (full details are given) and the authors state that the bond so made should be adequate for many purposes. (Replacement deposits of nickel, iron and zinc were less successful.) The bond obtained by the indium coating is, however, inferior to the strength of 50,000 p.s.i. obtained for nickel deposited on etched zirconium and subsequently alloy-bonded by diffusion treatment.

Hydrogen overvoltage on zirconium and a number of other metals is tabulated in the report.

Silver Plating of Nickel-Manganese Alloy Wire

'In-Plant Continuous Plating Line Silvers Wire Fastener.'

Iron Age, 1957, vol. 179, Apr. 11, pp. 122-3.

The article describes the plating installation set up by Wilbur Driver Company, Newark, N.J.,

* 6,000 p.s.i. for as-plated adhesion of nickel on etched zirconium.

for the silver plating of 0·036-in. diameter nickel-manganese wire. The system, which is completely automatic, comprises twelve cleaning, plating and rinsing operations, of which the following details are given:

Electrolytic treatment in a caustic-silicate solution: spray rinsing; electropickling in sulphuric-acid solution (65 per cent. by vol.); spray rinsing; nickel strike in a nickel-chloride solution; spray rinsing; dip in an aqueous solution of potassium cyanide; silver-cyanide dip; silver plating from cyanide solution; spray rinse; soaking rinse in hot water; drying in a forced-air system. The cycle described gives the soundness and quality of silver deposits essential to ensure a satisfactory coating on the wire when it is subsequently drawn down to 0·002–0·007-in. diameter.

Electrodeposited Nickel-Zinc Alloys

E. RAUB and F. ELSER: 'The Structure of Electrodeposited Alloys. XI. Nickel-Zinc Alloys.'
Metalloberfläche, 1957, vol. 11, May, pp. 165–8.

The authors briefly review the findings of earlier investigators with regard to the influence of small amounts of zinc on the deposition characteristics of nickel-plating solutions and on the properties of nickel deposits. Particular reference is made to the work carried out under the auspices of the American Electroplaters' Society Research Project.

The object of the experimental determinations now reported was to ascertain the composition of the alloys which would be electrodeposited, under varying conditions, from zinc-containing nickel-base electrolytes, and the influence of the zinc constituent on the deposition potential of nickel. The structure of the alloys deposited was also examined and correlated with the constitutional diagram of the binary system. Watts-type and chloride solutions were used and the effect of adding p-toluol sulphonamide to the Watts bath was also studied.

In deposition from the zinc-containing baths there was strongly preferential deposition of zinc. Current density, temperature of the solution and degree of agitation all proved to be highly significant factors in relation to the composition of the alloys deposited.

Tin-Nickel Plating on Paint Brushes

'Tin-Nickel Plated Brush Ferrule.'
Tin and its Uses, 1957, No. 39, p. 12.

A photograph shows a paint brush incorporating a tin-nickel-plated copper ferrule. After fourteen months of continuous service by a professional house painter using polyvinyl acetate emulsion paint the plating on the ferrule showed no sign of deterioration.

Properties of Thin Films of Nickel-Chromium-Alloy

See abstract on p. 118.

Electroless Nickel in Semi-Conductor Devices

M. V. SULLIVAN and J. H. EIGLER: 'Electroless Nickel Plating for Making Ohmic Contacts to Silicon.'
Jnl. Electrochemical Soc., 1957, vol. 104, Apr., pp. 226–30.

The use of silicon in semi-conductor devices is reported to be growing very rapidly. Each of these devices requires at least two electrical connexions to the silicon, but it has not been found possible to make simple and reliable soft-solder joints direct, as could be done in the case of germanium. Various types of contact have been proposed to overcome this difficulty, but none has proved entirely successful. Certain alloy-type contacts are in some respects satisfactory, but exposure to the temperatures involved in fabrication usually results in a loss of the minority charge carrier lifetime of the silicon. In some applications this degradation of life is undesirable.

The present authors (who are associated with Bell Telephone Laboratories, Inc.) report that, of the various methods studied for making contact at low temperature, an electroless nickel plating appears to be outstanding, from both a mechanical and an electrical standpoint. The method of plating is that developed by Brenner, and is already being used for a number of purposes, but the application now reported is believed to be novel.

The composition of the bath used is shown below:—

	g./L.
Nickel Chloride ($NiCl_2.6H_2O$) ..	30
Sodium hypophosphite ($NaH_2PO_2.H_2O$)	10
Ammonium citrate [$(NH_4)_2HC_6H_5O_7$]	65
Ammonium chloride (NH_4Cl) ..	50

After filtering, ammonium hydroxide (NH_4OH) was added until the solution turned from green to blue (pH 8-10).

This hot alkaline plating bath, with its rapid evolution of hydrogen, has excellent cleaning properties, and in many cases no special treatment of the silicon surface is necessary to obtain an adherent nickel plating. In the experiments reported, however, consistency and reproducibility of data were ensured by a uniform cleaning treatment, details of which are given in the paper.

Determination of the thickness of plating deposit as a function of time, at a temperature of 95°±2°C. (203°±4°F.), showed that the initial rate of deposition was of the order of 0·02 mm. nickel per hour, but that after about half an hour the rate dropped to about 0·005 mm. per hour. (These data are in reasonably good agreement with the value of 0·008 mm. per hour reported by Brenner.)

A semi-quantitative evaluation of the adhesion of the electroless nickel to silicon was made by measuring the force required to pull from the surface a copper wire which had been soldered to the nickel.

Optimum adherence was obtained with a plate thickness of about 0·002 mm. on a lapped surface and 0·001 mm. on a polished surface. In thicker films there was some loss of adherence, probably due to the increase of internal stress in the nickel plating. Electrical measurements recorded in the paper demonstrated the suitability of the nickel coatings for use on both *n* and *p* types of silicon.

In addition to the properties referred to above, other characteristics are noted as being of interest in connexion with the use of electroless nickel, e.g., the hardness of the coating as deposited, the additional hardening which occurs on heat-treatment, and the phosphorus in the nickel. Further advantages include the fine-grained laminated structure of the deposit, its brightness, and its relative freedom from porosity, also the possibility of uniform deposition over all shapes. A similar type of deposit may be used on germanium.

Nickel Flashing Prior to Enamelling

J. P. FINLEY: 'A Study of Nickel Deposition.'

International Enamelist, 1957, vol. 6, Apr., pp. 15, 19.

This paper records the results of experiments made to determine the conditions optimum for large-scale nickel flashing of steel range-liner parts on which a grey-oxide ground coat is used. The report includes an account of tests in which various factors were investigated as affecting the thickness and quality of the nickel deposit and the time required for deposition treatment. The influence of time, temperature of plating, pH, and composition of the solution was studied. The conclusions from the respective tests are individually recorded.

As a result of this work, and of tests carried out to ascertain the thickness of nickel required to ensure adequate adhesion of the enamel coatings, the following specification was set up:

> Thickness of coating 0·02–0·03 g/sq. ft., obtained by plating from a solution containing 1 oz./gal. of single nickel salts, operated at 150°–155°F. (65°–68°C.). It has been found possible to work with such a solution for about two weeks without addition of sodium hypophosphite, but for the remainder of the life of a batch of solution about one pound of sodium hypophosphite is used per day, added in two equal amounts: one at the beginning of an 8-hour shift and the other after 4 hours of operation. Near the end of the life of a batch of solution a larger amount of sodium hypophosphite must be added at the beginning of each working week. The total useful life of each batch is of the order of three months, after which time the bath becomes loaded with sludge, even when continuously filtered.

Nickel Overlay on Paper-Press Rolls

See abstract on p. 110.

Measurement of Thickness of Nickel Plating

G. D. LINSEY: 'Nickel Depth Meter.'

U.S. Atomic Energy Commission, Report H.W.-44720, July 31, 1956; 18 pp.

The report describes, with photographs and drawings, an electronic instrument based on eddy-current principles, permitting measurement of the thickness of thin layers of nickel plated on a non-magnetic base metal. Circuits are described for a particular model, which is capable of measuring thickness of nickel plating over a range of 0·1 to 2·0 mils, with an accuracy of about 5 per cent.

Determination of Copper in Nickel-Plating Solutions

T. A. DOWNEY: 'The Determination of Copper in Nickel Solutions.'

Plating, 1957, vol. 44, Apr., pp. 383–5.

The procedure proposed is based on a sensitive specific colorimetric method, details of which have been given in earlier literature (*Analytical Chemistry*, 1952, vol. 24, p. 371; 1953, vol. 28, p. 1588; 1954, vol. 26, p. 577).

The method is based on the formation of a yellow-coloured complex produced by the interaction of cuprous copper and neo-cuproine (2-9 dimethyl 1-10 phenanthroline). The yellow metallo-organic complex is extracted into chloroform and the absorbancy of the chloroform extract is measured. The absorbancy value is then converted, by reference to a previously standardized curve, to milligrams of copper present in the sample.

An adaptation of this procedure has proved suitable for estimation of copper in plating solutions: the experiments made in this connexion are described in the present note, and working instructions are given. Among the advantages of the method are that the reagents required are stable, and the procedure is specific and simple. The use of chloroform, which is heavier than the nickel solution, simplifies the extraction of the copper/neo-cuproine complex. The sensitivity of the method is such that it is possible to determine one mg. of copper in a litre of nickel solution, with an accuracy closer than ±5 per cent. A further point of practical significance is that because the copper/neo-cuproine complex in the ethanol-chloroform mixture is quite stable, it is not necessary to determine the absorbance of the solution immediately.

NON-FERROUS ALLOYS

Nickel-Copper Smelters and Nickel Refineries in Canada

CANAD. DEPT. OF MINES AND TECHNICAL SURVEYS, OTTAWA: 'Metallurgical Works in Canada. Part II. Non-Ferrous and Precious Metals.'

List I-1, Jan., 1957; 21 pp. Price 25 cents.

The information contained in this publication covers the location, capacity, products and ownership

of smelters and/or refineries producing aluminium, antimony, bismuth, cadmium, calcium, cobalt, copper and brass, lead, magnesium, nickel, precious metals, selenium, tantalum, tellurium, titanium, tungsten, uranium and zinc. Copper and brass rolling mills are also included.

Thermal Expansion of High-Nickel Alloys

P. HIDNERT: 'Thermal Expansion of Some Nickel Alloys.'

Jnl. of Research, Nat. Bur. Standards, 1957, vol. 58, Feb., pp. 89–92; *R.P.* 2737.

This paper contains a summary of data obtained, between 1918 and 1956, in the laboratories of the National Bureau of Standards: it may, therefore, be regarded as a reference document.

The table on pp. 116–117 gives a comprehensive review of the values found. All the determinations were made by means of the micrometric thermal-expansion apparatus described in an earlier paper (*N.B.S. Circular* 486, 1950.)

A series of curves illustrates points of particular interest, including observations on Inconel alloys, and a correlation of the thermal-expansion coefficients of nickel-chromium alloys with and without additions of other elements. Some of the main comments made are noted below:

The 'annealed' samples of Inconel, containing 0·02–0·11 per cent. of carbon, were probably in a supersaturated condition with respect to carbon, as a result of relatively fast cooling from 2050°F. (1121°C.). On first heating of these alloys during the determination of thermal expansion, irregularities were observed, at about 500° and 700°C., in the curves of instantaneous coefficients of expansion *vs.* temperature. The effect at 500°C. has been attributed (in an unpublished communication) to the formation of an ordered structure based on Ni_3Cr, but the author considers that there is inadequate evidence for such a conclusion. Irregularities at the higher temperature, 700°C., are believed to be caused by precipitation of carbide: a similar effect, in nickel-chromium-iron alloys, had been previously reported by the present author, who had noted that these effects were observed only during the first heating, on alloys which had previously been cast or quenched from a high temperature. The magnitude of the irregularity observed in Inconel increased with rise in carbon content, within the range present in the three alloys studied. No irregularities were observed in the curves of Inconel on cooling from 1000°C. to room temperature, and the author considers that the expansion curves of each sample, on repeated slow heating and slow cooling, will closely follow the curve obtained on first cooling from 1000°C. to room temperature.

Observations on Monel (at temperatures up to 1000°C.) show that the expansion was not reversible on heating and cooling the samples during the first test, but that reversibility was obtained on the second test. Comparison of the values for Monel with those found for nickel indicates that the addition of 27–29 per cent. of copper to nickel increases the coefficients of expansion by a maximum of $1{\cdot}4 \times 10^{-6}$ per °C., i.e., by 10 per cent.

The presence of appreciable amounts of other elements, e.g., as in the complex alloys Waspalloy and Illium, results, over various ranges between 20° and 700°C., in coefficients of thermal expansion appreciably lower than those of nickel.

Correlation of N.B.S. values and other data on nickel-chromium alloys, with and without small additions of other elements, for the range 20° (or 0°C.) to 100°C., shows that coefficient of expansion increases slightly with rise in chromium content. The presence of 11 per cent. of cobalt or 7 per cent. of copper, with about 10 per cent. of other elements, appreciably reduced the coefficients of expansion of nickel-chromium-base alloys.

High-Temperature Tensile Properties of Nickel, Copper-Nickel Alloys and Copper

W. D. JENKINS, T. G. DIGGES and C. R. JOHNSON: 'Tensile Properties of Copper, Nickel, 70 per cent. Copper–30 per cent. Nickel and 30 per cent. Copper–70 per cent. Nickel Alloys at High Temperatures.'

Jnl. of Research, Nat. Bur. Standards, 1957, vol. 58, Apr., pp. 201–11; *R.P.* 2753.

This paper is one of a series reporting investigations of the rheological behaviour, at sub-zero and at elevated temperatures, of high-purity nickel and copper and alloys of these two metals. Reports on the earlier sections of the investigation are listed in the *bibliography* to the present paper, which deals specifically with the influence of temperature on the short-time tensile properties of the materials mentioned above.

Tests were made at temperatures ranging from 70° to 1500° or 1700°F. (21° to 815° or 926°C.), on the materials in the annealed condition and after cold-drawing to 40 per cent. reduction. Properties at sub-zero temperatures, taken from previous reports, are included for comparison. In addition, tensile tests were made at various temperatures on the 30–70 nickel-copper alloy as cold-drawn to 25 and to 70 per cent. reduction in area, and on copper after annealing to produce a relatively large grain size.

The results of these tests, and of determination of the properties of other alloys in the copper-nickel system, show that the tensile properties of the materials examined are closely dependent on the percentage of solute atoms in the component lattices, the temperature of test, and the degree of cold working. The strengthening influence of the presence of solute atoms was at a maximum in the temperature range in which strain-ageing occurred (300°–700°F.: 150°–370°C.), but some effect was evident at all temperatures of test.

The increase in yield and tensile strengths, in relation to the amount of nickel in the alloys, was greater in the range 0–30 per cent. nickel than in the range 30–70 per cent. The effects on ductility, of the solute atoms present, varied appreciably with

Sample	Commercial Name	Chemical Composition *a*									Treatment	Diameter or cross section of sample
		Ni	Cr	Cu	Fe	C	Mn	Si	S	Other Elements		
		%	%	%	%	%	%	%	%	%		mm.
1260	Nickel *c*	99·94	—	0·006	0·03	0·005	—	0·006	0·004	Co, 0·016	Cold-swaged and annealed at 950°C.	4
462	Manganese nickel	97·0*e*	—	0·3	0·8	—	1·6	—	—	—	Drawn to wire	2
1587 *f*	Hoskins alloy 667 *g*	94·5	—	—	—	0·04	3·9	0·81	—	—	Cast in metal mold, hot-rolled to ¼-in. diameter and cooled in air.	6
1859	Inconel *h*	76·45	14·96	0·15	7·89	0·07	0·26	0·19	0·007	—	Hot-rolled and machined to 12-mm. diameter, annealed by rapid insertion into preheated furnace at 2,050° F. (1,121° C.), held at this temperature for 9½ min. and then quickly removed from furnace and cooled in quiescent air.	12
1858	do. *h*	75·99	14·42	0·22	8·87	0·02	0·28	0·17	0·007	—	do. do.	12
1860	do. *h*	75·64	15·32	0·19	8·17	0·11	0·33	0·21	0·007	—	do. do.	12
1790	Evanohm *i*	75	20	2·5	—	—	—	—	—	Al, 2·5	Drawn to wire	
571 *j*	Monel metal	67·6	—	26·7	—	—	—	—	—	—	Hot-rolled	1
889 *k*	do.	66·80	—	29·20	1·70	0·24	2·00	0·06	—	—	—	1
1861 *j*	do.	66·4	—	29·4	—	—	—	—	—	—	Annealed 3 hr. at 1,000°C. in pure dry H_2 and furnace-cooled (150°/hr. from 1,000° to 600° and then 85°/hr. to 20°.	1
908 *m*	do.	66·2	—	29·3	—	—	—	—	—	—	—	1
572	do.	—	—	—	—	—	—	—	—	—	Hot-rolled	
1054	M-M-M Alloy *n* (Modified Monel metal)	61	—	25	3·5	—	0·75	0·75	—	Sn, 9	Cast in sand mold	16
1032 *p*	Illium alloy	59	24	7	—	—	1·5	1·5	—	Mo, 4 W, 2 Ag, 1	Cast	
1862	Waspalloy *q*	55·45	19·22	0·12	0·73	0·45	0·67	0·47	0·008	Co, 11·20 Mo, 7·00 Ti, 2·49 Al, 1·03 P, 0·015	Annealed 3 hr. at 1,000°C. in pure dry H_2 and furnace-cooled (150°/hr. from 1,000° to 600° and then 85°/hr to 20°).	

(a) All compositions are given in per cent. by weight.

(b) H indicates that the coefficients of expansion were obtained on heating, and C indicates that they were obtained on cooling the samples.

(c) Added for comparison with the nickel alloys in this paper.

(d) Determined by P. Hidnert and reported by L. Jordan and W. H. Swanger, The Properties of Pure Nickel, B.S. J. Research 5, 1291 (1930) RP257. The coefficients of expansion for other temperature ranges were computed from the original observations.

(e) Includes cobalt, probably as much as 1 per cent.

(f) Chemical composition determined by W. H. Jukkola, formerly of the Bureau, and by J. L. Hague of the Chemistry Division of the Bureau.

(g) Used in the manufacture of spark plugs and may be classed as a heat-resistant alloy.

(h) Made by International Nickel Co. Chemical composition determined by this company.

Diameter or cross section of sample	Test No. *b*	Average Coefficients of Expansion per Degree										
		20° to 60°C.	20° to 100°C.	20° to 200°C.	20° to 300°C.	20° to 400°C.	20° to 500°C.	20° to 600°C.	20° to 700°C.	20° to 800°C.	20° to 900°C.	20° to 1000°C.
mm.		×10⁻⁶	×10⁻⁶	×10⁻⁶	×10⁻⁶	×10⁻⁶	×10⁻⁶	×10⁻⁶	×10⁻⁶	×10⁻⁶	×10⁻⁶	×10⁻⁶
4	1H	—	13·3*d*	13·9	14·4*d*	15·0	15·2	15·5*d*	15·7	16·0	16·3*d*	—
2	1H	—	—	14	—	15	—	15	—	16	—	—
6	1H	—	13·2	13·7	14·3	14·5	14·9	14·9	15·0	15·3	15·6	16·0
	1C	—	13·1	13·6	14·3	14·7	15·1	—	—	—	—	17·0
	2H	—	13·0	13·7	14·3	14·7	15·1	15·4	15·9	16·2	16·6	17·0
12	1H	—	13·3	13·8	14·2	14·6	14·9	15·3	15·8	16·0	16·3	16·8
	1C	—	13·2	13·8	14·2	14·6	15·0	—	—	—	—	17·4
12	1H	—	13·2	13·7	14·1	14·5	14·8	15·3	15·8	16·2	16·6	17·0
	1C	—	13·2	13·8	14·2	14·6	15·0	15·5	—	—	—	17·3
12	1H	—	13·3	13·8	14·2	14·5	14·9	15·4	15·8	16·0	16·3	16·9
	1C	—	—	—	14·1	14·5	14·9	15·4	15·9	16·3	—	17·3
1	1H	13	—	—	—	—	—	—	—	—	—	—
10	1H	—	14·3	14·8	15·1	15·5	—	—	—	—	—	—
	1C	—	14·5	15·0	15·3	15·8	—	—	—	—	—	—
11	1H	—	—	14·7	—	15·5	15·7	—	—	—	—	—
13	1H	13·7*l*	13·9	14·5	14·9	15·4	15·8	—	—	16·9	—	17·6
	2H	—	14·0	14·6	15·0	15·4	15·8	16·2	—	17·0	—	—
	2C	—	—	—	—	—	—	16·3	16·6	17·0	—	—
11	1H	—	—	14·6	—	15·3	15·5	—	—	—	—	—
10	1H	—	14·2	14·8	15·2	15·6	—	—	—	—	—	—
	1C	—	14·7	15·3	15·5	15·9	—	—	—	—	—	—
16 by 21	1H	—	—	14·2	—	15·1	16·0*o*	—	—	—	—	—
	1C	14·2	—	—	15·2	15·5	16·4	—	—	—	—	—
10	1H	—	12·5	13·0	13·5	—	—	—	—	—	—	—
13	1H	—	12·4	12·8	13·2	13·6	13·9	14·6	15·1	15·8	16·8	17·9
	2H	—	12·4	12·8	13·1	13·6	—	—	—	—	—	—
	2C	—	12·4	12·8	13·1	13·6	—	—	—	—	—	—

(i) Made by Wilbur Driver Co., Newark, N.J.

(j) Chemical composition determined by E. E. Maczkowske of the Chemistry Division of the Bureau.

(k) Cut from valve stem. Chemical composition determined by H. A. Buchheit, formerly of the Bureau.

(l) One-third of the average coefficient of cubical thermal expansion ($41 \cdot 0 \times 10^{-6}$ per deg. C. from 4° to 66°C.) determined on another sample by a volumetric method, by C. T. Collett and B. C. Keysar of the Mechanics Division of the Bureau.

(m) Cut from valve stem. Chemical composition determined by E. E. Maczkowske of the Chemistry Division of the Bureau.

(n) Resistant to acid and erosion by high-temperature, high-pressure steam.

(o) P. Hidnert and W. T. Sweeney, Thermal expansion of M-M-M alloy, Phys. Rev. **36**, 787 (1930).

(p) The Standard Calorimeter Co., East Moline, Ill., reported specific gravity 8·3 and melting point 1,300°C.

(q) Made by Universal Cyclops Steel Corp., Titusville, Pa. This alloy resists corrosion, has structural strength at high temperatures, and is used for rotor blades in turbojets.

(*reproduced by courtesy of the author and of the National Bureau of Standards*)

temperature of test, degree of cold drawing and the criterion of ductility used. The effects of cold drawing were diminished, or entirely removed, at temperatures above the re-crystallization temperature.

At a given temperature, the 70 per cent. nickel alloy was appreciably stronger than the 30 per cent. nickel type.

Properties of Nickel-Aluminium Alloys

See abstract on p. 130.

Solubility of Hydrogen in Nickel Alloys

'The Solubility of Hydrogen in Alloys.'

K. H. LIESER and G. RINCK: 'II. The Nickel-Zinc System.'

Zeitsch. f. Elektrochemie, 1957, vol. 61, Apr., pp. 357-9.

W. SIEGELIN, K. H. LIESER and H. WITTE: 'III. Study of the Ternary Systems $MgCu_2$-$MgAl_2$, $MgCu_2$-$MgSi_2$, $MgNi_2$-$MgCu_2$ and the Binary Systems Ag-Cd, Cu-Be, Ag-Mg, Cu-Mg, Ni-Al, Ni-Si, Co-Al, Fe-Al.'

ibid., pp. 359-66.

K. H. LIESER and H. WITTE: 'IV. Discussion of the Results.'

ibid., pp. 367-76.

(*Note*: Part I of this series, dealing with the systems $MgNi_2$-$MgZn_2$, appeared in *Zeitsch. f. physikalische Chemie*, 1954, vol. 202, pp. 321 et. seq.)

Titanium-Carbide/Nickel System

W. N. EREMENKO, et al: 'The Interaction of Titanium Carbide and Nickel.' Problems of Powder Metallurgy and Strength of Materials: *Akad. Nauk Ukrain. S.S.R.*, 1956, vol. 3, pp. 62-72.

Report of thermal analysis and metallographic and X-ray investigation, resulting in setting up of part of the TiC-Ni system, on the nickel-rich side. The main conclusions are reported in *Metallurgical Abstracts*, 1957, vol. 24, Apr., p. 627.

Grain Growth of Titanium-Carbide in Nickel

L. P. SKOLNICK: 'Grain Growth of Titanium Carbide in Nickel.'

Trans. Amer. Soc. Metals, 1957, vol. 209, pp. 438-42; *T.P.* 4406E.

Jnl. of Metals, Apr., 1957, Sect. II.

Properties of materials containing a hard constituent dispersed in a metallic matrix are dependent on the distribution of the hard phase: some study of the variables affecting distribution has already been made, on tungsten-carbide/cobalt, nickel/titanium-carbide and other systems. In the research reported in this paper grain growth of titanium carbide in nickel was investigated in order to elucidate the microstructural development characteristics of this and analogous systems. The infiltration method was used for preparation of the specimens: the advantages of this technique, as compared with that of sintering, are pointed out.

Three successive stages of growth were observed: (1) formation of idiomorphic grains, (2) spheroidization, accompanied by rapid growth, and (3) a period of coalescence and closer packing of the carbide grains. It is concluded that the primary mode of grain growth is by dissolution of high-energy surfaces, transport of carbide through the liquid phase, and isothermal re-deposition.

Properties of Thin Films of Nickel-Chromium Alloy

R. H. ALDERSON and F. ASHWORTH: 'Vacuum-deposited Films of Nickel-Chromium Alloy.'

Brit. Jnl., Applied Physics, 1957, vol. 8, May, pp. 205-10.

The work reported relates to thin films of nickel-chromium alloys deposited under carefully controlled conditions by evaporation of 80-20 nickel-chromium alloy from a tungsten 'boat' strip, by resistance heating. The substrate was borosilicate heat-resisting glass.

The properties of the alloy films discussed include composition in relation to the temperature of the source, the relationship between the thickness of the film and its electrical resistance, temperature coefficient of resistance, grain size, mechanical failure of stressed films, optical transmission as a function of film thickness, fundamental noise, and electrical breakdown under low- and high-frequency conditions. The data are summarized in a series of graphs, and photomicrographs illustrate grain-growth, breakdown due to stresses in the film, and electrical breakdown in microwave attenuators.

Corrosion-Resisting Applications of Monel and Nickel

See abstract on p. 143.

Copper-Nickel Casting Alloy for Marine Use

G. L. LEE: 'New Casting Alloy Combines High Strength and Corrosion-Resistance.'

Modern Castings, 1957, vol. 31, May, pp. 74-9.

Successful results, in the marine field, with the wrought 90-10 copper-nickel alloy containing small amounts of iron and manganese has been followed by the development of a companion casting alloy, which gives good resistance to corrosion and erosion in sea-water conditions. This paper describes some of the experiments and tests which were carried out in developing the cast material, and gives an account of its properties.

It has been found that an alloy containing 12 per cent. of nickel, with aluminium 1·5, manganese 1, and iron 0·6, per cent., deoxidized with 0·05 per cent. each of phosphorus, titanium and calcium (as calcium-silicon), will show a tensile strength of 95,000 p.s.i. (42·5 tons per sq. in.), and that it is suitable for pressure castings and structural parts for use under severe marine conditions. Another cast copper-base alloy, containing nickel 12, manganese 1, iron 1·2, per cent. gives a tensile strength of

45,000 p.s.i. (20 tons per sq. in.), and can be used for pressure castings. Corrosion tests in sea water have shown both alloys to be superior to the commonly used bronzes.

In discussion of production practice, it is recommended that ample gating and risering be ensured, together with prevention of turbulence. It is essential that lead be kept out of all silicon-deoxidized bronzes; if 0·1 per cent. or more of lead is present hot-shortness and cracking occurs.

Influence of Surface Films on Friction and Wear

See abstract on p. 107.

Resistance of Nickel-containing Aluminium Alloys to Distilled Water

See abstract on p. 145.

Brazing of Copper-Nickel Alloys

W. H. MUNSE and J. S. ALAGIA: 'Strength of Brazed Joints in Copper Alloys.'

Welding Jnl., 1957, vol. 36, Apr., pp. 177S-84S.

The investigation reported was concerned with various factors affecting the strength of brazed joints in copper alloys: main emphasis was on the strength of the brazing materials. The basis materials used were copper-silicon, copper-nickel (70-30 and 90-10) and aluminium-bronze alloys. Lap joints, both with and without fillets, were made with these materials, and four representative filler metals were tested: for compositions see below. In addition, butt joints of each of the base alloys were made, using only the BAg-1 brazing alloy: see table below.

A detailed study was made of the effect of basis metal, filler metal, type of joint, and brazing procedure. From the results it is confirmed that fabrication procedure is of vital importance in securing sound brazed joints in these copper alloys. On the basis of the experimental results reported it is concluded that 'the lengths of lap necessary to develop the full tensile strengths of the basis alloys with sound joints would be approximately as shown in the table below'.

Electrodes for Arc Welding of Monel and Nickel

F. A. BALL and D. R. THORNEYCROFT: 'Aspects of the New Monel and Nickel Arc-Welding Electrodes.'

Reprint from *Welding and Metal Fabrication*, Feb., 1957; see abstract in *Nickel Bulletin*, 1957, vol. 30, No. 4, pp. 57-8.

Issued by MOND NICKEL CO., LTD.

Welding of Nickel-Aluminium-Bronze Propellers

J. L. CAHILL: 'Welding of Nickel-Aluminium Bronze.'

Reprint of paper from *Welding Jnl.*, 1956, vol. 35, Dec., pp. 1226-30; abstracted in *Nickel Bulletin*, 1957, vol. 30, No. 3, p. 39.

Issued by INTERNATIONAL NICKEL CO., INC.

Filler Metals used in Brazing Copper Alloys

A.W.S./ A.S.T.M. Designation	Composition					Solidus Temperature	Liquidus Temperature	Brazing Range
	Cu %	Ag %	Zn %	Cd %	P %			
BAg-1	15	45	16	24	—	1125°F. 607°C.	1145°F. 618°C.	1145°-1440°F. 618°-782°C.
BAg-6	34	50	16	—	—	1270°F. 687°C.	1425°F. 774°C.	1425°-1600°F. 774°-871°C.
BCuP-5	80	15	—	—	5	1185°F. 640°C.	1500°F. 815°C.	1300°-1500°F. 704°-815°C.
BCuP-4	87	6	—	—	7	1185°F. 640°C.	1380°F. 748°C.	1300°-1500°F. 704°-815°C.

(It is noted that the BCuP filler metals were not used with the copper-nickel alloys.)

	Lap Length, in Multiple of Thickness			
	BAg-1	BAg-6	BCuP-5	BCuP-4
Copper-silicon	2·07	1·96	1·73	2·23
Copper-nickel (70-30)	1·53	1·66	—	—
Copper-nickel (90-10)	1·35	1·38	—	—
Aluminium-bronze	2·63	2·45	2·08	2·54

Spectrochemical Analysis of Nickel Alloys: Specifications

AMER. SOC. TESTING MATERIALS: 'Report of Committee E-2 on Emission Spectroscopy.'
A.S.T.M. Preprint 55, June 1957; 70 pp.

This report includes the following items which are of interest in relation to nickel-containing materials:

(1) 'Report on Co-operative Tests on Proposed Tentative Method for Spectrochemical Analysis of Nickel Alloys by the Powder-D.C. Arc Technique.' *Appendix I*, pp. 5–7.

To test the accuracy of the method proposed as E-2 S.M. 5-1 (see '*Methods for Emission Spectrochemical analysis*' published by the Society in 1948), five spectrochemical laboratories and nine chemical laboratories made independent tests on two National Bureau of Standards nickel-oxide standards. The results, given in this report, are considered to indicate a sufficiently high degree of accuracy and reproducibility to justify issue of the E-2 S.M. 5-1 procedure as Tentative Recommended Practice.

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(2) 'Survey regarding Sizes and Shapes of Graphite Electrodes.'*Appendix II*, pp. 8–10.

Survey of current practice in spectrochemical laboratories shows that 29 different types of electrode will meet all requirements. A Proposed Tentative Recommended Practice for Designation of Shapes and Sizes, of Preformed Graphite Electrodes has, therefore, been drawn up (pp. 66–70 of the report).

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(3) P. V. MOHAN and T. P. SCHREIBER: 'Suggested Method for Spectrochemical Analysis of Nickel-base High-Temperature Alloys by the Point-to-Plane Spark Technique.' E-2 S.M. 5–10; pp. 17–20.

Essential features of the method

Point-to-plane controlled spark excitation is employed. Nitrogen is blown into the analytical gap. Intensity ratios of analytical lines and nickel internal standard lines are obtained from photometric readings and a plate-calibration calculating board. Concentrations are obtained from analytical curves relating log intensity ratio to log concentration.

The method covers analysis of nickel-base high-temperature alloys, for the following elements, in the percentage concentration ranges indicated:

Chromium (13·0–18·0), iron (7·0–13·0), molybdenum (4·0–6·0), aluminium (2·0–4·0), titanium (0·6–3·0), silicon (0·25–1·0), manganese (0·02–0·80), boron (0·001–0·07).

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(4) F. R. POTTER: 'Suggested Method for Spectrochemical Analysis of Aluminum and its Alloys by the Point-to-Plane Spark and Intermittent-Arc Techniques, using a Recording Photoelectric Spectrometer.' E-2 S.M. 7–10; pp. 27–39.

The method recommended provides for determination of 23 elements, over various concentration ranges, including nickel (0·001–10·0 per cent.).

(5) E. J. HUNEMORDER and K. R. SCHMECK: 'Suggested Method for Spectrochemical Analysis of Magnesium and its Alloys by the Pin-to-Pin Spark Technique.' E-2 S.M. 7–11; pp. 40–4.

The method provides for determination of 10 elements, in varying concentrations, including nickel (0·0004–0·10 per cent.).

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(6) J. H. McCLURE and R. E. KITSON: 'Suggested Method for the Spectrochemical Analysis of Cobaltic Oxide for Impurities by the Powder-D.C. Arc Technique.' E-2 S.M. 11–00, pp. 56–60.

The steps involved in this method of analysis were described in *Analytical Chemistry*, 1953, vol. 25, pp. 867–8. The sample, in the form of a dry powder ground to pass a No. 200 (74-micron) sieve, is arced, using standard conditions in a centre-post-type graphite electrode. A step sector is used with each exposure, spectral lines are photographed, and intensity ratios of selected analytical lines to cobalt internal standard lines are determined photometrically. The A.S.T.M. schedule covers determination of 9 elements, in varying concentrations, including nickel (calculated as NiO) over the range 0·03–3·0 per cent.

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(7) 'Proposed Tentative Method for the Spectrochemical Analysis of Nickel Alloys by the Powder-D.C. Arc Technique', pp. 61–5.

This method previously appeared as Suggested Method E-2 S.M. 5-1 in '*Methods for Emission Spectrochemical Analysis*', issued by the Society in 1948.

The sample is dissolved in dilute nitric acid and evaporated to dryness. The powder is then mixed with graphite powder, which acts as a buffer and diluent. This mixture is packed into the crater of a graphite cup. Direct-current arc excitation is employed. The spectra are recorded photographically. Intensity ratios of selected pairs of analytical lines and internal standard nickel lines are determined photometrically. The concentration of each element is read from an analytical curve relating the log intensity ratio to log concentration.

The procedure may be used for determination of the following elements in nickel alloys, in the percentage concentrations shown:

Cobalt (0·01–1·0), copper (0·001–0·30), iron (0·005–0·30), magnesium (0·005–0·30), manganese (0·005–0·30), aluminium (0·003–0·30), chromium (0·003–0·10), lead (0·003–0·10), silicon (0·003–0·10), titanium (0·003–0·10), zinc (0·003–0·10), boron (0·003–0·010).

Although primarily designed for inspection testing of nickel-alloy thermionic cathodes, the method is stated to be equally applicable to the determination of the elements listed when present in any alloy in which the nickel content is greater than 98·0 per cent., provided that the total sample available is more than 50 mg.

Determination of Nickel in Aluminium Alloys

J. A. DEAN and C. CAIN: 'Flame Spectrophotometric Determination of Copper, Nickel and Manganese in Aluminum-base Alloys.'

Analytical Chemistry, 1957, vol. 29, Apr., Pt. I, pp. 530–2.

Copper, nickel and manganese can be selectively extracted with chloroform, as the metal diethyldithiocarbamates, from a buffered aqueous solution containing citrate. The elements can then be successively determined by aspirating the chloroform extract into an oxyacetylene flame. This use of an organic reagent in conjunction with extraction simultaneously separates several constituents of the sample from the matrix prior to the determination. The method described obviates many spectral and radiation interferences encountered when the elements are determined in an aqueous solution. In addition, the chloroform enhances the emission of each of the elements. When this procedure is used for aluminium-base alloys only cobalt interferes.

Determination of Copper in Nickel-Plating Solutions

See abstract on p. 114.

NICKEL-IRON ALLOYS

Dynamic Magnetostrictive Properties of Nickel-Iron Alloys

C. M. DAVIS, H. H. HELMS and S. F. FEREBEE: ' Dynamic Magnetostrictive Properties of Ni-Fe Alloys.'

Jnl. Acoustical Soc. of America, 1957, vol. 29, Apr., pp. 431–4.

In 1944 Pan investigated the magnetic and magnetostrictive properties of 45 per cent. nickel nickel-iron alloys and observed a maximum electromechanical coupling coefficient of approximately 0·29, i.e., a value 0·94 per cent. of the maximum reported for nickel. In view of the wide range of nickel-iron alloys now commercially available, and the characteristics which had been found in the 45 per cent. nickel alloy, an investigation was undertaken, at the U.S. Naval Ordnance Laboratory, to determine the potential usefulness of nickel-iron alloys as active elements in magnetostrictive transducers. The range covered was 35–67·5 per cent. nickel. The materials, in the form of toroids made from ring laminations, were evaluated by the motional impedance method. Annealing temperatures varied from 600°C. to 1200°C., and the effect of various annealing techniques was investigated. The report includes data on measured values of the electromechanical coupling coefficient, reversible permeability, dynamic magnetostrictive constant and other parameters.

The electrochemical coupling coefficient for alloys containing from 40 to 52·5 per cent. of nickel was greater than 0·31; a maximum value of 0·37 was found for the 40 per cent. nickel alloy. The product of the magnetostrictive constant and the reversible permeability for the above range of alloys was greater than 95×10^4 (dynes/oersted-cm^2) and reached a maximum value of approximately 130×10^4 for the alloy containing approximately 40 per cent. of nickel.

Stacking Faults in Magnetic Nickel Alloys

R. D. HEIDENREICH and E. A. NESBITT: 'Stacking Disorders in Nickel-base Magnetic Alloys.'

Physical Rev., 1957, vol. 105, Mar. 1, pp. 1678–9.

Short note on observations made on Perminvar, Permalloy and cobalt-nickel alloys. Results to date indicate that stacking faults in single-phase magnetic alloys may be quite important as affecting response to heat-treatment in a magnetic field. A fuller account of the work is to be published later.

CAST IRON

S.G. Iron in Tankers

M. PARIS and B. DE LA BRUNIÈRE: 'The Corrosion-Resistance of Ductile Iron in Sea Water and Petroleum-Tanker Services.'

Corrosion, 1957, vol. 13, May, pp. 292t–5t.

The information presented in this paper is based on laboratory experiments carried out at the Research Centre of La Société des Fonderies de Pont-à-Mousson, and on service data relating to piping commercially produced in France and installed in tankers. The purpose of the paper is to compare the corrosion-resistance of grey cast iron, S.G. iron, and steel, as exhibited in sea and distilled waters, in some common acid and alkaline corrosive solutions, and in corrosive media encountered in petroleum-tanker service.

The results confirm general experience that S.G. iron has approximately the same corrosion-resisting characteristics as grey cast iron, and that in most conditions its behaviour is superior to that of steel.

The reports on piping in tankers show that after five years' service S.G. iron pipes are still in good condition and that corrosion attack appears to have completely stopped. In similar conditions steel pipes always required replacement.

Steam-heating coils carrying steam at 150°C., which have been operating for periods varying up to four years, have been found to be entirely intact, with no trace of serious deterioration, whereas steel connecting pipes in the same system have had to be removed, due to severe corrosion.

It is noted that 40 tankers are now equipped with S.G. iron piping.

Nickel-Iron Electrodes for Repair Welding of Marine Engineering Components

H. C. WAUGH: 'Welding Repair Costing $600 Reclaims $8,000 Engine Blocks.'

Reprint of paper from *Welding Jnl.*, 1956, vol. 35, Aug., pp. 801–3; abstracted in *Nickel Bulletin*, 1956, vol. 29, Nos. 10–11, p. 187.

Issued by INTERNATIONAL NICKEL CO., INC.

CONSTRUCTIONAL STEELS

Effect of Alloying Elements on Breakdown of Martensite

A. S. KENNEFORD and T. WILLIAMS: 'Effect of Some Common Alloying Elements on the Breakdown of Martensite in a W.Q. 0·35 per cent. Carbon Steel.' *Jnl. Iron and Steel Inst.*, 1957, vol. 185, Apr., pp. 467–74.

The effect of various alloy elements, on the breakdown of martensite, was studied on water-quenched 0·35 per cent. carbon steels heated to, and cooled from, about 550°C. at a rate of 2·5°C./min. Changes in length were determined by means of a Leitz universal dilatometer permitting measurements of very small variations.

The steels used were in form of ⅝-in. diameter bar, forged from small h.f.-furnace melts. Eight series were examined:

		%	
(1)	Carbon steels containing	0·3—1	Carbon
(2)	Steels containing silicon	0·5—3	Carbon in all cases about 0·35 per cent.
(3)	„ „ manganese	0·5—2·5	
(4)	„ „ nickel	0·5—5	
(5)	„ „ chromium	0·5—3	
(6)	„ „ molybdenum	0·25—1	
(7)	„ „ vanadium	0·05—0·3	
(8)	„ „ cobalt	0·5—4	

The observations made indicate that nickel, manganese, chromium, molybdenum, vanadium and cobalt have little effect on the temperature range over which martensite decomposes into ferrite and cementite. Chromium, molybdenum and vanadium delay softening during tempering, an effect which, according to the literature, is due, in the case of chromium to inhibition of spheroidization of cementite, and with molybdenum and vanadium to secondary hardening caused by formation of complex carbides. Of all the elements studied, silicon is the only one which delays tempering by raising the temperature at which martensite decomposes into ferrite and cementite. This effect has considerable practical significance in connexion with the production of high-strength steels resistant to tempering.

Influence of Alloy Elements on High-Temperature Tensile Strength of Normalized Steel

J. GLEN: 'Effect of Alloying Elements on the High-Temperature Tensile Strength of Normalized Low-Carbon Steel.'

Jnl. Iron and Steel Inst., 1957, vol. 186, pp. 21–48.

The report is based on tests made on a series of steels containing varying amounts of the following elements: manganese, chromium, molybdenum, tungsten, vanadium, titanium, silicon, nickel and copper. The effects of the alloy elements were individually studied, and some of the steels were also made both with and without additions of aluminium.

It was found that for a simple iron-carbon alloy one strain-age-hardening effect was obtained, which was at a maximum at about 200°C. When a little nitrogen was present this strain-age-hardening effect was intensified.

Under the experimental conditions employed, no strain-hardening effect was observed in steels alloyed with vanadium or titanium, and it was also found that the alloying elements modified the effects due to carbon and nitrogen.

The nickel-steel series studied comprised compositions representative of a 3 per cent. type and two austenitic steels containing, respectively, 31·2 and 41·6 per cent. of nickel. In these steels, and in a 1 per cent. silicon steel, the alloy elements had little or no effect on strain-hardening characteristics.

Corresponding to each strain-age-hardening effect at a certain temperature, a minimum in reduction of area was also obtained. It was concluded that the maxima in stress and the minima in ductility were related to some form of coherent precipitation of carbides in the dislocations.

The occurrence, in the simple austenitic nickel steels, of a strain-hardening maximum in stress and a minimum in the reduction of area at about 200°C. suggests that such phenomena are not confined to ferritic steels.

Elevated-Temperature Properties of Medium-Carbon Nickel-Alloy Steels

W. F. SIMMONS and H. C. CROSS: 'Report on Elevated-Temperature Properties of Wrought Medium-Carbon Alloy Steels.'

A.S.T.M. Special Tech. Publn. No. 199; 123 pp. Published by the Society, 1957. Price $4·25.

This is one of a series of reports on the elevated-temperature properties of various types of material, prepared and issued under the auspices of the A.S.T.M./A.S.M.E. Joint Committee on Effect of Temperature on the Properties of Metals. (Earlier reports are listed and an outline is given of other projects on which information will be published in the near future.)

The data presented (in tabular and graphical form) have been obtained from research laboratories, commercial publications and the technical literature: sources are given on each data sheet. The steels to which reference is made are divided into groups according to the principal alloying elements (see p. 123), and information is given on the heat-treatment which the respective specimens have undergone. The report includes summary curves showing tensile strength, 0·2 per cent. offset yield strength, percentage elongation and reduction of area; stresses for rupture in 100, 1,000, 10,000 and 100,000 hours, and stresses for creep rates of 0·0001 and 0·00001 per cent. per hour (1 per cent. in 10,000 and in 100,000 hours).

Twenty-seven steels are dealt with, representing approximately twelve types of medium-carbon alloy steel, and the report also includes data on a

few miscellaneous low-carbon alloy steels. The groups of steel covered are shown below.

Cr-Mo
Cr-Mo-V
Cr-Mo-W
Cr-Ni
Cr-Ni-Mo
Cr-Ni-Mo-V
Cr-Ni-Mo-W-V
Cr-W-V
Cr-V
Ni-Mo-V

Miscellaneous Alloy Steels
Nitralloy G
Nickel Steel to S.A.E. 2335 specification
Manganese Steel (low-alloy)
Tungsten Steel (low-alloy)

Miscellaneous Low-Carbon Alloy Steels
Mayari R
Carilloy T-1
N.A.X. AC 9115
A.M.S. 5602
Timken D. M.

Heat-Treatment of Nickel-Alloy Steel for Prevention of Hair-Line Cracking

C. R. GARR and A. R. TROIANO: 'Flaking of Heavy Alloy-Steel Sections.'
Trans. Amer. Inst. Mining and Metallurgical Engineers, 1957, vol. 209, pp. 445-9; *T.P.* 4379E:
Jnl. of Metals, Apr., 1957, *Sect. II.*

There is now general agreement that the presence of hydrogen in one or more forms is a pre-requisite for the occurrence of flaking (hair-line cracking) in alloy steels. Laboratory research recently reported has, however, demonstrated that hydrogen alone will not cause the conventional type of flaking: a combination of transformation stresses and hydrogen is required. If, therefore, a steel section can be heat-treated in such a manner as to ensure elimination of transformation stresses flaking will be prevented, irrespective of the hydrogen content of the steel.

The aim of the investigation now reported was to provide evidence which would answer the following questions:

(1) Is it possible, by extension of the laboratory results, to develop an anti-flaking heat-treatment suitable for commercial practice ?

(2) If steel sections previously subjected to anti-flaking treatment are subsequently treated by conventional methods, will hair-line cracks be produced ?

The material used was a commercially produced low-nickel-chromium-molybdenum steel into which an unusually high content of hydrogen (10 cc. per 100 kg.) had been introduced by injecting steam into the heat after completion of the oxidizing period and again after introduction of manganese and chromium. Details of production methods and heat-treatment are given.

Treatment consisting primarily in an isothermal transformation in the pearlitic range produced flake-free steel forgings, in spite of extremely high hydrogen content. Cycles in the bainite range were not, however, successful in preventing flaking, primarily because a hundred per cent. isothermal transformation in this range was not attained.

Flake-free steel was subjected, some six to nine months later, to further treatment consisting in austenitizing for one hour at 1750°F. (955°C.), with subsequent air-cooling. Examination of the sections after ageing for one month showed that this treatment had caused severe flaking, indicating that the anti-flaking cycle had not conferred immunity under subsequent unsuitable heat-treatment.

Crack Propagation in Hydrogen-Induced Static Fatigue

W. J. BARNETT and A. R. TROIANO: 'Crack Propagation in the Hydrogen-Induced Brittle Fracture of Steel.'
Trans. Amer. Inst. Mining and Metallurgical Engineers, 1957, vol. 209, pp. 486-94; *T.P.* 4285E.
Jnl. of Metals, Apr., 1957, *Sect. II.*

The time-dependent phenomenon of delayed failure or static fatigue has recently been the subject of numerous investigations, the most significant result of which has been the recognition of the rôle of hydrogen as a cause of embrittlement. The occurrence of delayed failure by embrittlement has in most cases been limited to steels at strength levels above 180,000 p.s.i. (80 tons per sq. in.): it has been most prevalent in material which has been subjected, during processing, to an environment conducive to absorption of hydrogen, and it has been possible to produce static-fatigue failure in high-strength steels into which hydrogen had been purposely introduced by cathodic charging or other means. Static-fatigue failure manifests itself, however, in a more cataclysmic manner than does the usual loss of ductility associated with hydrogen embrittlement and may even occur when the conventional tensile-ductility criterion reveals no apparent embrittlement.

Following a brief summary of conclusions reached by Frohmberg *et al.*, in a comprehensive study of hydrogen-induced static fatigue and embrittlement in a low-alloy nickel-chromium-molybdenum steel at three ultra-high strength levels, the present author reports a further investigation on steel from the same heat, the main object being to analyse the nature of the static-fatigue failure process. Fracture was studied with respect to the occurrence and mechanism of initiation of cracking and crack propagation. The steel was tested at three tensile levels: 270,000; 240,000 and 230,000 p.s.i. (120·5; 108 and 102·5 tons per sq. in.). A high hydrogen content was introduced into the surface layers by means of cathodic charging.

An electric-resistance method, of which full details are given, was developed for analysis of the kinetics of crack growth. The authors' summary of their findings is as follows:

'The mechanism of static fatigue fracture of

high-strength steels has been identified as a process of hydrogen-induced crack initiation and propagation, climaxed by catastrophic fracture. On application of a load in excess of the static fatigue limit a crack formed at the root of the circumferential notch and propagated radially inward. The initiation process in general was independent of time, although the possibility of an induction period does exist under certain conditions which may involve a relatively homogeneous distribution of hydrogen. The static fatigue life at a given applied stress was determined by the crack propagation and fracture behaviour.

'The state of hydrogen distribution produced by cathodic charging, and the subsequent redistribution on ageing, determined the crack-propagation and fracture characteristics.

'For the charging conditions employed, a relatively thin case of high hydrogen content resulted. On subsequent ageing, inward diffusion increased the depth of hydrogen penetration. The depth of the hydrogen-containing case and the hydrogen-concentration gradient existing at the initiation of crack growth determined the kinetics of crack propagation. Propagation through the hydrogen-rich case was relatively fast and appeared to be controlled by a local microscopic diffusion process. This has been termed the first stage of crack growth. The kinetics of the second stage of crack propagation, growth beyond the pre-existent case, were controlled by the macroscopic diffusion of hydrogen. If fracture followed second-stage crack growth, the core ahead of the advancing crack was essentially free of hydrogen and exhibited a fracture strength which was characteristic of the hydrogen-free material.

'Crack propagation appeared to be dependent on some critical combination of stress and hydrogen concentration. A mechanism based on the strain-induced trapping of hydrogen is proposed as a tentative explanation of the observed effect of pre-cracking on subsequent static-fatigue behaviour and the effect of stress on the rate of second-stage crack growth.'

Stepwise Austenitizing of Nickel-Alloy Steel

E. P. KLIER, V. WEISS and G. SACHS: 'Stepped Austenitizing Treatment for 4340 Steel.'

Trans. Amer. Inst. Mining and Metallurgical Engineers, 1957, vol. 209, pp. 424–5; *T.N.*383E. *Jnl. of Metals*, Apr., 1957, *Sect. II.*

It has been shown that the isothermal transformation of austenite in a special steel can be modified by isothermal holding in the high sub-critical transformation range (*ibid.*, 1955, vol. 203, p. 707). Since this treatment potentially leads to increased hardenability, tests have been made to determine whether the stabilization effects observed can be used with advantage on nickel-chromium-molybdenum steel of the S.A.E. 4340 type. Jominy hardness tests were made on specimens 2·5 in. in diameter and 8 in. long, and, in view of the fact that the steel studied is much used at strength levels at which notch-sensitivity is of major concern, this property was evaluated by notch-tensile tests on specimens of several sizes.

The stepped austenitizing treatment was found to promote deeper hardening in this steel, while at the same time giving reduced notch-sensitivity in the high-strength range in which the material is now being used.

Heat-Treatment of Low-Alloy Nickel-Chromium-Molybdenum Steels

D. J. BLICKWEDE and R. C. HESS: 'Heat-Treatment Diagram for A.I.S.I. 4340.'

Metal Progress, 1957, vol. 71, Apr., p. 96B.

(C 0·43, Mn 0·76, Ni 1·85, Cr 0·81, Mo 0·20, per cent.).

'Heat-Treatment Diagram for A.I.S.I. 9840.'

ibid., May, p. 96B.

(C 0·43, Si 0·25, Mn 0·84, Ni 1·00, Cr 0·81, Mo 0·23, per cent.).

Fatigue-Resistance of Ultra-High-Tensile Steel

P. W. RAMSEY and D. P. KEDZIE: 'Prot Fatigue Study of an Aircraft Steel in the Ultra-High-Strength Range.'

Trans. Amer. Inst. Mining and Metallurgical Engineers, 1957, vol. 209, pp. 401–6; *T.P.* 4395E. *Jnl. of Metals*, Apr., 1957, *Sect. II.*

In view of the critical nature of the cyclic loads to which aircraft structures are subject in service, much interest centres on the fatigue properties of ultra-high-tensile steels recently developed for use in that field. One of the types showing greatest promise for such applications is a nickel-chromium-molybdenum-vanadium composition, to S.A.E. (A.M.S.) specification 6434, for which the following limits are laid down: carbon 0·31–0·38, silicon 0·20–0·35, manganese 0·60–0·80, nickel 1·65–2·00, chromium 0·65–0·90, molybdenum 0·30–0·40, vanadium 0·17–0·23, per cent. Phosphorus and sulphur are each limited to 0·040 per cent. maximum.

The work reported in this paper was carried out to assess the fatigue properties of this steel in the heat-treated conditions giving, respectively, tensile strength levels of 155,000; 180,000; 195,000; 225,000; 240,000; 260,000 and 280,000 p.s.i. (70, 80·5, 87, 101·5, 107, 116 and 125 tons per sq. in.).

The tests were made by the Prot procedure (*Rev. Métallurgie*, 1948, vol. 45, pp. 481–9), which has the advantage (by comparison with the conventional Wöhler method) of reducing the number of specimens required and of shortening the test period. The Prot test consists of progressively increasing cyclic stress until failure occurs. By varying the rate of load, α, a useful relationship is found to exist with failure stress, so that for steel the failure stress appears to be a linear function of $\alpha^{\frac{1}{2}}$. By extrapolating this line to the ordinate a value of failure stress, called the Prot endurance limit, is obtained.

This can also be considered as the failure stress for an infinitely slow rate of loading, which corresponds to Wöhler test conditions ($\alpha=0$). A typical test set-up is illustrated in the paper.

The following conclusions are drawn from the results, which are individually reported for the steels at all the tensile levels mentioned.

> In carefully ground specimens of this type of steel there is a useful increase in fatigue strength with rising tensile strength, up to about the 240,000 p.s.i. tensile level, with an endurance ratio of 0·40–0·42. Above this tensile strength the fatigue strength appears to level off or even to drop slightly.

In almost all cases fatigue failure started at a non-metallic inclusion, indicating the importance of cleanliness in relation to fatigue strength.

The Prot method appears to be a useful test technique for high-strength aircraft steels, and at the 195,000 p.s.i. tensile strength level the results correlated well with those obtained by the Wöhler method.

Influence of Thickness of Plate on Properties of Pressure-Vessel Steels

J. H. GROSS and R. D. STOUT: 'Properties and Weldability of High-Strength Pressure-Vessel Steels in Heavy Sections.'

Welding Jnl., 1957, vol. 36, Mar., pp. 157S–67S.

In a previous investigation the mechanical properties and weldability of six high-strength steels were studied, with the object of assessing their suitability for use in pressure vessels designed to higher-than-conventional stress levels (*ibid.*, 1956, vol. 35, pp. 115S–19S: *Nickel Bulletin*, 1956, vol. 29, No. 6, pp. 108–9). The results of these tests proved encouraging and led to the extension of the work to plate of greater thickness (4-in.). These further tests are now reported.

The examination covered study of the effects of plate thickness and of position in the plate, on the mechanical properties and weldability of the following types of steel:

(1) Carbon steel to A.S.T.M. A-201-54T: Grade A firebox quality.

(2) Low-carbon manganese-molybdenum steel to A.S.T.M. A-302-54T: Grade B firebox quality.

(3) Navy G steel: a low-carbon steel containing approximately 2·5 per cent. of nickel with 0·55 per cent. of molybdenum.

(4) T-1 steel, a low-alloy nickel-chromium-molybdenum-vanadium type.

Full details are given of the results of tests made to determine the tensile, Charpy-impact and fatigue qualities of the steels, also their impact-transition temperatures and their welding characteristics. Metallographic and hardness surveys were included in the investigation. The data obtained are shown in comparison with relevant information on 1-in. plate.

The following conclusions are drawn:

(1) The 0·505-in. tensile properties differed appreciably between the 4-in. and 1-in. thicknesses only in Navy G and T-1 steels. No important differences were observed in any of the steels as a function of location in the 4-in. plate.

(2) The 4-in. full-section tensile properties were very similar to those obtained with the 0·505-in. test piece, except for a loss in ductility introduced by the difference in the geometry of the specimen.

(3) In elevated-temperature tensile tests the carbon steel showed considerable strain-ageing: for the higher-strength steels the loss in ductility was relatively less. In all cases the behaviour of the plate of the two thicknesses was similar.

(4) In the V-notch Charpy tests the carbon steel was the least tough. In all the steels the effect of position in the plate was negligible. The change from 1-in. to 4-in. plate improved the notch-toughness of the manganese-molybdenum steel, lowered that of the Navy G and the T-1 types, and left essentially unchanged that of the carbon steel.

(5) The notch-toughness of the 4-in. plate in the welded condition was inferior to that found for the 1-in. plate. Stress-relief treatment brought the four heavy-section steels to more nearly the same level of transition temperature.

(6) Underbead cracking was appreciable only in the manganese-molybdenum and the T-1 steels: the tendency to cracking was considerably less in the 4-in. than in the 1-in. plates.

(7) The plastic fatigue strengths of all four steels were found to be approximately proportional to their tensile strengths: only the 4-in. carbon steel and 1-in. nickel-chromium-molybdenum-vanadium steel deviated significantly from this relationship.

(8) No appreciable variation in hardness or microstructure was detectable through the thickness of the 4-in. specimens, but exhaustive study would be required to establish fully the relationship of properties to microstructure.

A Quarter-Century's Progress in the Nickel Industry

See abstract on p. 108.

Nickel-containing Steels for Pressure Vessels

'Tentative Specification for Chromium-Copper-Nickel-Aluminum Alloy Steel Plates for Pressure Vessels',
contained in Report of A.S.T.M. Committee A-1 on Steel.
A.S.T.M. Preprint 1, June, 1957; pp. 27–9.

The steel covered is a type giving tensile strength in the intermediate range, and is specially intended for applications in which welding is needed. It may be made by open-hearth or electric-furnace procedure, and when the plates are intended primarily for sub-zero service the steel should preferably be deoxidized in accordance with approved practice

for production of fine grain. Heat-treatment is preferably normalizing.

The composition limits are as under:

		%
Carbon		0·12 max.
Silicon	Ladle	0·10–0·35
	Check	0·08–0·37
Manganese	Ladle	0·55–1·00
	Check	0·50–1·05
Sulphur		0·040 max.
Phosphorus ..		0·040 max.
Nickel	Ladle	0·50–0·95
	Check	0·47–0·98
Chromium	Ladle	0·50–0·95
	Check	0·44–1·01
Copper		0·40–0·75
Aluminium		0·04–0·30

Tensile and bend requirements are specified.

Nickel-Alloy Steel for Aircraft Bolts

BRIT. STANDARDS INSTN.: 'Low-Nickel-Chromium Steel.'

*B.S. Specification S.*122, Apr., 1957.

The specification covers hardened-and-tempered bars for the manufacture of hot-forged bolts of $\frac{1}{2}$ in. to $1\frac{1}{2}$ in. shank diameter.

Composition limits are as follows:

	%
Carbon	0·30—0·40
Silicon	0·10—0·35
Manganese	0·6 —0·9
Nickel	1·0 —1·5
Chromium	0·45—0·75
Sulphur	⩾0·050
Phosphorus	⩾0·050

The mechanical properties in the heat-treated condition are specified as:

0·1 per cent. Proof Stress	⩽43 tons per sq. in.
Ultimate Tensile Stress	54–56 tons per sq. in.
Elongation	⩽18 per cent.
Izod Impact	⩽40 ft.-lb.

Nickel Steel for Rivets

AMER. SOC. TESTING MATERIALS: 'Tentative Specification for High-Strength Structural Alloy Rivet Steel', contained in report of A.S.T.M. Committee A-1 on Steel.

A.S.T.M. Preprint 1, June, 1957, pp. 12-13.

The specification relates to alloy rivet steel suitable for use with material furnished in accordance with A.S.T.M. Designation A-242 for High-Strength Low-Alloy Structural Steel, and equivalent steels.

The compositional limits called for are shown in the table in the right-hand column.

Tensile properties are specified, and an upsetting test is called for.

	Ladle Analysis %	Check Analysis %
Carbon, max.	0·08	0·10
Silicon ..	0·7 —1·0	0·64—1·06
Manganese ..	0·25—0·50	0·21—0·54
Nickel ..	0·70—1·00	0·67—1·03
Copper ..	0·50—0·75	0·48—0·77
Aluminium ..	0·10—0·30	0·08—0·32

Nickel-containing Electrodes for Production of High-Strength Welds

D. C. SMITH: 'Ultra-High-Strength Weld Metal with Low-Hydrogen Electrodes.'

Welding Research Council Bull. No. 36, May, 1957; 14 pp.

A rapidly increasing variety of ultra-high-strength steels is now in supply or under development in several countries, and there is urgent need for arc-welding electrodes which will be capable of depositing weld metal giving tensile strength, ductility and toughness equal to, or better than, that of the parent metals. In an introductory section the author of this report discusses some of the views held with regard to the most promising lines for development of ultra-high-strength steels and considers some of the problems involved. A detailed account is then given of work done in the Electrode Division of the Harnischfeger Corporation, with the object of devising electrode compositions which would (*a*) be suitable for welding ultra-high-tensile steels already in commercial supply and (*b*) be satisfactory for use on other steels of that type currently under investigation. The compositions typical of steels in the two groups are shown on page 127.

Some 150 compositions were studied, from the point of view of their ability to develop ultra-high-strength weld metal when subjected to standard heat-treatment procedures. The materials investigated were tested by determination of transverse and all-weld-metal tensile and yield strengths, ductility, hardness and toughness, hardenability, bending properties, and behaviour under restrained-cracking tests. Typical compositions and tensile strengths of the six types of core wire recommended are shown on page 127.

The only type of coating found suitable in any of the experimental electrodes was the low-hydrogen type. A lime type coating (i.e., one containing less than 8 per cent. TiO_2), developed for use with iron-powder low-hydrogen electrodes, had the most suitable operational characteristics, and this type was used in development of all the six varieties of weld metal referred to above.

Surface Treatments for Use on Low-Alloy Steels

See abstract on p. 112.

Ultra-High-Tensile Steels being used in Production Aircraft (U.S.A., 1955)

Name or Type	S.A.E./ A.M.S.*	Average Composition							Approximate Tensile Strength	
		C %	Si %	Mn %	Ni %	Cr %	Mo %	V %	p.s.i.	t.s.i. (approx.)
4340 ..	6415	0·40	†	0·75	1·83	0·80	0·25	—	270,000	120
4340 modified	6427	0·30	†	0·90	1·83	0·85	0·43	0·08	250,000	112
H.S. 220 ..	6407	0·30	0·55	0·70	2·05	1·20	0·45	—	230,000	103
Hy-Tuf ..	6418	0·25	1·50	1·35	1·83	0·30	0·40	—	230,000	103

* Aeronautical Materials Specification (S.A.E.). † Normal silicon 0·20-0·35 per cent.

Ultra-High-Strength Steels under Investigation (U.S.A. 1955)

Name or Type	C %	Si %	Mn %	Ni %	Cr %	Mo %	V %	B %	Tensile Strength	
									p.s.i.	t.s.i. (approx.)
4350 ..	0·50	†	0·75	1·83	0·80	0·25	—	—	300,000	134
H.S. 260 ..	0·40	0·60	0·85	2·20	1·45	0·50	—	—	290,000	130
Super Hy-Tuf	0·40	2·30	1·30	—	1·40	0·35	0·20	—	300,000	134
High-C Super Hy-Tuf	0·47	2·42	1·28	—	1·11	0·42	0·25	—	325,000	145
98B40 modified	0·40	†	0·75	0·85	0·80	0·20	—	*	290,000	130
U.S.S. Strux	0·43	0·55	0·90	0·75	0·90	0·55	—	*	290,000	130
Tricent ..	0·43	1·60	0·80	1·83	0·85	0·38	0·08	—	300,000	134
Super Tricent	0·55	2·10	0·80	3·60	0·90	0·50	—	—	340,000	152
5% Cr Die Steel	0·40	†	0·80	—	5·0	1·30	0·50	—	268,000	115

† Normal silicon 0·20-0·35 per cent. * Boron present.

Weld-Metal Compositions

Type	C %	Si %	Mn %	Ni %	Cr %	Mo %	V %	Tensile Strength	
								p.s.i.	t.s.i. (approx.)
Mn-Cr-Ni-Mo ..	0·26	0·80	1·30	1·70	3·0	1·20	—	252,000	112
Mn-Cr-Ni-Mo-V ..	0·30	0·60	1·00	1·80	3·0	0·80	0·2	263,000	117
Cr-Ni-Mo	0·34	0·70	0·94	1·90	0·80	0·40	—	282,000	126
Cr-Mo	0·40	0·85	0·94	—	0·75	0·35	—	280,000	125
Si-Cr-Ni-Mo	0·35	1·61	0·84	1·55	0·74	0·34	—	275,000	123
Cr-Mo-V	0·40	0·94	0·69	—	5·0	1·50	0·5	285,000	127

Influence of Surface Films on Friction and Wear

See abstract on p. 107.

Determination of Non-Metallic Inclusions in Steels

C. W. SHORT, R. S. ROBERTS and G. CROALL: 'The Chlorine Method for the Determination of Non-Metallic Inclusions in Steel.'
Jnl. Iron and Steel Inst., 1957, vol. 186, May, pp. 85–9.

The authors direct attention to the comprehensive papers on this subject presented earlier by COLBECK (*Iron and Steel Inst. Special Reports Nos.* 16 *and* 25).

The present paper suggests some modifications in procedure, describes new apparatus for chlorinating the steel, and gives details of two separate schemes of analysis which may be employed. The accuracy of the chlorine technique is demonstrated by comparison of analytical results obtained by it with values given by vacuum-fusion methods. The steels analysed included a number of low-nickel types.

HEAT- AND CORROSION-RESISTING MATERIALS

Fatigue, Creep and Rupture of High-Temperature Materials: Reference Data

F. H. VITOVEC and B. J. LAZAN: 'Fatigue, Creep and Rupture Properties of Heat-Resistant Materials.' *Wright Air Development Center, Report* 56-181, Aug., 1956; 196 pp. *P.B.* 121,580.

This report is intended primarily to summarize data collected during extensive tests on the static and fatigue properties of typical high-temperature alloys. The scope of the tests made is shown below, and the compositions of the materials for which information is given are listed in the table.

Scope of Tests and Method of Reporting

A series of axial fatigue tests was made in a temperature range up to 1650°F. (898°C.), under various combinations of alternating and mean stress. Notched and un-notched specimens were used. The basic fatigue and creep data are presented, respectively, in S-N curves and in stress-range diagrams, to demonstrate the effect of stress-concentration and temperature on fatigue life and creep behaviour. Effective-stress-concentration data for notched specimens are also shown in diagrams. Relative effects of creep and of fatigue, on rupture, are discussed, as is the influence of temperature on these factors. Mechanical-property data typical of the alloys at room temperature are also included.

The following general conclusions are drawn from the experimental work recorded:

'In general there is no definite fatigue limit at high temperatures. In certain cases, however, primarily in notched specimens, the S-N curve is very flat.

'As would be expected, the static rupture strength falls more rapidly with rising temperature than does the fatigue strength.

'Stress concentration causes a decrease in fatigue strength and an increase in stress-rupture properties in the materials studied.

'Maximum notch-sensitivity does not always occur under reversed-stress conditions: in many cases the addition of a small static mean stress increases notch-sensitivity.

'The superposition of a relatively small alternating-stress amplitude to a given mean stress may either accelerate or decelerate creep strain, or may leave it unaffected.

'Mean stress as a governing factor in relation to creep becomes more significant with rise in temperature.'

Creep-Rupture Testing of Metals and Alloys

See abstract on p. 107.

Dynamic Elastic Modulus of Nickel-containing Materials at Elevated Temperatures

R. F. WILDE and N. J. GRANT: 'Dynamic Elastic Modulus Values at Elevated Temperatures for Nickel-base, Aluminum-base and Metal/Metal-Oxide Alloys.'
Amer. Soc. Testing Materials, Preprint 72, June, 1957; 9 pp.

The tests were made by a dynamic method designated the 'free-free' or 'floating-beam' procedure: for

Composition of Materials Tested

Alloy	C %	Mn %	Si %	S %	P %	Cr %	Ni %	Mo %	Co %	Ti %	Al %	Fe %	Other Elements
X-40 (Stellite 31) ..	0·46	0·76	0·71	0·009	0·011	25·88	10·62	—	Bal.	—	—	0·93	7·11 W
S-816	0·397	1·12	0·50	0·018	0·012	19·42	20·62	4·1	42·9	—	—	2·99	2·86 Nb 1·03 Ta 4·03 W
6·3% Mo-Waspalloy	0·136	0·56	0·31	—	—	19·68	53·3	6·32	13·55	2·59	1·04	2·5	—
7% Mo-Waspalloy ..	0·045	0·67	0·47	0·008	0·015	19·22	55·45	7·00	11·20	2·40	1·03	0·73	0·12 Cu
M-252	0·14	1·15	0·60	0·006	0·016	18·64	54·95	9·86	9·77	2·58	0·89	0·84	—
Timken 16-25-6 ..	0·102	1·92	0·528	0·009	0·01	16·45	27·0	6·60	—	—	—	Bal.	0·133 N_2
Inconel X-550 ..	0·05	0·73	0·28	0·007	—	14·92	Bal.	—	—	2·5	1·16	6·59	0·03 Cu 1·03 Nb +Ta
Crucible 422 ..	0·19	1·00	0·71	0·012	0·020	13·49	0·70	1·07	—	—	—	Bal.	1·02 W 0·30 V
Lapelloy	0·30	1·05	0·35	0·024	0·019	12·12	0·22	2·87	—	—	—	Bal.	0·28 V
Stainless 403 ..	0·12	0·51	0·34	0·010	0·018	12·20	0·06	0·05	0·03	—	0·009	Bal.	0·0089 Sn

description of the technique see *Jnl. Iron and Steel Inst.*, 1947, vol. 157, pp. 345-8 and *Metal Progress*, 1950, vol. 58, July, pp. 85-9, 96-100.

In the present paper the main characteristics of the method are outlined and a diagram of the apparatus is shown. Measurements were made on seven typical nickel-base high-temperature alloys, over the range 80°-1600°F. (27°-870°C.); on Type 316 (molybdenum-containing austenitic chromium-nickel steel) over the same temperature range; on pure aluminium and a number of high-purity solid-solution aluminium alloys from 80°-800°F. (27°-427°C.); on a number of sintered, extruded metal/metal-oxide alloys based on copper and on nickel, and on pure nickel (over various temperature ranges).

The nickel alloys for which the modulus was determined include Inconel X-550, Inco 700, Waspalloy, M-252 and three experimental alloys of nickel-chromium base containing varying amounts of aluminium and/or titanium. It was observed that at all temperatures the commercial nickel-base alloys showed a higher elastic modulus than the stainless steel, but that the curves are parallel. At temperatures of about 1400°-1600°F. (760°-870°C.) the stress for 100-hours' rupture life of the nickel alloys increases with increasing relative values of the elastic modulus. Wherever possible, comparable data from the literature are included, and, in connexion with the tests on extruded nickel, attention is called to earlier work by Köster (*Zeitsch. f. Metallkunde*, 1948, vol. 39, pp. 1-16), the results of which are partially confirmed by the present authors.

High-Temperature Tensile Properties of Nickel, Copper-Nickel Alloys and Copper

See abstract on p. 115.

Resistance of Austenitic Steel to Thermal Fatigue

E. E. BALDWIN, G. J. SOKOL and L. F. COFFIN: 'Cyclic-Strain Fatigue Studies on A.I.S.I. Type 347 Stainless Steel.'
Amer. Soc. Testing Materials, Preprint 64, June 1957; 15 pp.

The importance of resistance to thermally induced stresses is increasingly recognized as one of the factors determining the suitability of a material for service in nuclear-power plants, power-producing equipment such as gas- and high-temperature steam turbines, and other plant in which rapid temperature changes are liable to occur. In view of the preference for the austenitic steels for many parts incorporated in such plant, it has become desirable to build up reference information on their thermal-fatigue characteristics.

In this paper test results are reported for a stainless steel subjected to constant-temperature cyclic strains ranging from 0·0035 in. per in. to 0·02 in. per in., where pronounced non-linearity between cyclic stress and cyclic strain occurs and where failures result in from 10^3 to 5×10^5 cycles. The influence of the following variables was investigated: temperature (600°C. max.), grain size of the steel, anisotropy, and sequence of loading.

Effect of Thermal Treatment and Structure on Properties of Nimonic 80A

W. BETTERIDGE and A. W. FRANKLIN: 'The Effect of Heat-Treatment and Structure on the Creep and Stress-Rupture Properties of Nimonic 80.'
Jnl. Inst. Metals, 1956-57, vol. 85, July, pp. 473-9.

Investigation of the constitution of the Nimonic series of alloys, correlated with empirically determined effects of varying cycles of heat-treatment, has indicated that optimum creep properties in such materials are not developed by a simple process of precipitation hardening. It was found by FLOYD and TAYLOR, in their study of the nickel-chromium-aluminium ternary system, that the precipitating phase of the type $Ni_3(Ti,Al)$, which they designated γ', is completely dissolved in the matrix at temperatures above about 875°C., and, when precipitated by heating the alloy at lower temperatures, produces hardening. The creep and stress-rupture properties of the alloy at about 700°C. were, however, found to be optimum when a solution-treatment temperature of about 1080°C. was used. Lower temperatures resulted in higher rates of creep, and higher temperatures caused early rupture, at small values of creep strain.

The investigation reported in the present paper was undertaken to study these effects more fully. The first section of the report describes preliminary experiments made to obtain a clearer understanding of the phenomena influencing the properties of the alloy; the second contains the results of a series of tests made to assess the effects of various heat-treatments on creep properties. Since the preliminary investigations had indicated that the precipitation of chromium carbide represents an important structural change occurring at temperatures in the region of the normal solution-treatment temperature, a systematic study was made of the effects of such precipitation, on the creep and rupture properties of the alloy. The work comprised (1) determination of the effect of treatments at temperatures between 1150° and 700°C., after solution treatment at 1250°C.; (2) determination of the effect of various treatments in the range 1000°-700°C., after precipitation of chromium carbide to an approximately optimum degree, and (3) study of the effect of variation in the solution-treatment temperature, when followed by given precipitation treatments. The criterion used was the creep properties of the alloy at 17 tons per sq. in. and 750°C.

The data presented indicate that three factors control the creep characteristics of Nimonic 80A :

(1) The time and temperature of solution-treatment largely determine the creep rate. Temperature has the predominant effect.

(2) Precipitation of chromium carbide at the grain boundaries largely determines the amount of creep extension which occurs before fracture.

(3) The time and temperature of precipitation of the normal hardening phase, $Ni_3(Ti,Al)$, affects, to a lesser degree, both creep rate and extension at fracture.

The two-stage treatment normally used for this

alloy provides a reasonable compromise in properties. The temperature of solution-treatment is high enough to ensure a reasonably low creep rate, and is suitable also for the deposition, at the grain boundaries, of carbide taken into solution by earlier heating for hot working. Precipitation at 700°C. alone leads to a low creep rate and a long life, but the use of a higher temperature for the precipitation treatment, although involving some sacrifice in those properties, makes it possible to obtain increased creep extension before fracture.

A Quarter-Century's Progress in the Nickel Industry

See abstract on p. 108.

Thermal Stability of Age-Hardened Alloys

A. W. COCHARDT: 'Some Estimates of the Thermal Stability of Dispersion-Hardened Alloys.'

Trans. Amer. Inst. Mining and Metallurgical Engineers, 1957, vol. 209, pp. 434–7; *T.P.* 4415E.
Jnl. of Metals, Apr., 1957, *Sect. II.*

The service temperatures at which many of the precipitation-hardened alloys are used are often in the ranges in which over-ageing and structural changes may occur, and, in consequence, the strength of the alloys, although initially high, may fall somewhat rapidly in course of time, resulting in relatively short service life. The question of the thermal stability of high-temperature alloys is thus of primary importance, and it is the object of this paper to discuss factors which govern that property. An attempt is also made to estimate the time which will produce over-ageing of some of the hardener constituents most commonly used in nickel- and iron-base high-temperature alloys. To explain the mechanism of over-ageing, the author takes as typical the intermetallic compound Ni_3Al, which is the principal hardener in such alloys as Inconel X 550, Inconel 700, Superior Waspaloy and some other high-temperature alloys. It is, however, pointed out that the actual over-ageing process is considerably more complicated than the simple case considered.

An estimation is made of the time in which structural changes should occur in alloys hardened with Ni_3Al, Al_2O_3, Fe_3C, TiC and TiN. It was found that particles of the intermetallic, the carbide, or the nitride, (of 200Å radius), would dissolve at 1340°F. (726°C.) in a nickel-base matrix within minutes or hours, whereas the size of the oxide particle would not change appreciably within several hundred years.

Influence of Titanium and Aluminium on Creep Properties of Nickel-base High-Temperature Alloy (Waspaloy)

D. I. SINIZER: 'Investigation of Nickel-base Precipitation-Hardening Alloys.'

Wright Air Development Center, Tech. Report 55-218, Sept., 1955; 38 pp. *P.B.* 111,791.

The work reported was carried out to determine the influence of variation in titanium and aluminium content on Waspaloy alloy, for which the specification limits laid down by the makers (Pratt and Whitney Corporation) were, at the time of the tests, as shown below:

Carbon 0·10 max., manganese 1·00 max., sulphur 0·03 max., silicon 0·75 max., chromium 18·00–21·00, cobalt 12·00–15·00, molybdenum 2·50–3·50,* titanium 2·20–2·80, aluminium 1·00–1·50, iron 2·00 max., copper 0·50 max., per cent., nickel balance.

The research was designed also to extend general knowledge on the stress-rupture behaviour of vacuum-melted nickel-base precipitation-hardening alloys of the types falling within such specification.

The alloys examined were made with closely controlled carbon content and all were within the limits shown above. Standard melting and casting procedures were found to be satisfactory, except that an unusually large hot top appears to be necessary in order to ensure sound ingots. All the ingots made forged satisfactorily.

The tests showed that increasing the titanium/aluminium ratio from about 1·5 to 3·0 had no appreciable effect on rupture life or minimum creep rate at 1500°F. (815°C.), for stresses from 30,000 to 35,000 p.s.i. (13·4 to 15·6 tons per sq. in.). At 52,500 p.s.i. (23·45 tons per sq. in.) and 1350°F. (732°C.) none of the alloys were notch-sensitive.

Pre-Precipitation Structures in Nickel-Chromium-base Alloys

J. MANENC: 'X-Ray Investigation of Structural Development in a 'Modified' 80–20 Nickel-Chromium Alloy.'

Rev. Métallurgie, 1957, vol. 54, Mar., pp. 161–8.

X-ray studies on precipitation-hardening nickel-chromium-base alloys containing titanium and aluminium confirm that, prior to actual precipitation of the hardening constituent, the homogeneity of the solid-solution matrix is perceptibly disturbed. This change in structure, already revealed by other methods of study, causes abnormal effects in the X-ray lines: typical examples are illustrated in the paper. The author advances an explanation for the effects observed.

Properties of Nickel-Aluminium Alloys

E. M. GRALA: 'Investigation of the NiAl Phase of Nickel-Aluminum Alloys.'

Nat. Advisory Committee for Aeronautics, Tech. Note 3828, Jan., 1957; 33 pp.

Study of the effects of composition and of homogenization heat-treatment, on the hardness and tensile properties of cast alloys of the NiAl intermetallic phase. This phase exists over a wide range of composition (at 500°C. from approximately 24 to 37 weight per cent. aluminium), with stoichiometric NiAl at 31·5 per cent. aluminium.

Relatively small changes in composition within the NiAl phase resulted in appreciable variations in hardness and strength. Room-temperature hardness

* Later raised to 3·5-5·0 per cent.

of alloys containing 25–35 per cent. of aluminium exhibited a sharp minimum at the stoichiometric composition, and the average room-temperature tensile strength of as-cast alloys fell, with increasing aluminium content, from 22,300 p.s.i. (10 tons per sq. in.) for the 25 per cent. aluminium alloy to 14,250 p.s.i. (6·4 tons per sq. in.) for the 31·5 per cent. alloy. Homogenization of the cast alloys resulted in no large changes in room-temperature tensile strength, and neither the as-cast nor the homogenized alloys showed any measurable ductility at room temperature.

At 1500°F. (815°C.) the tensile strength of homogenized alloys also fell with increasing aluminium content, ranging from 29,050 p.s.i. (13 tons per sq. in.) for the 25 per cent. alloy to 14,500 (6·5 tons per sq. in.) for the 31·5 per cent. alloy. In these high-temperature tests alloys containing up to 31·5 per cent. of aluminium showed a considerable degree of ductility.

Additions of 0·5–2·0 per cent. of molybdenum to the 31·5 per cent. aluminium alloy refined the grain size and significantly increased both room-temperature and elevated-temperature strength and ductility.

High-Temperature Properties of Complex Nickel-Molybdenum-base Alloys

O. PRESTON, N. J. GRANT and C. F. FLOE: 'Final Report: Development and Testing of Vacuum-Melted Nickel-Molybdenum Alloys with Minor Alloying Additions.' *U.S. Atomic Energy Commission, Oak Ridge Nat. Laby., Report O.R.N.L.* 2181; 37 pp.

In an earlier report covering the examination of some binary nickel-molybdenum alloys (molybdenum 17–36 per cent.) and a Hastelloy alloy it was recorded that the severe embrittlement which occurred at high temperatures in the binary alloys was not found in the more complex Hastelloy alloy.* The work now published was carried out to determine the degree of stabilization which would be conferred on two of the intermediate binary alloys by means of additions of chromium, iron, vanadium and niobium.

* See Nickel Bulletin, 1957, vol. 30, No. 6, p. 99.

The compositions of the alloys examined are shown in the table below:

The examination comprised:

(1) Creep-rupture tests, up to about 500 hours, at 1350°, 1500° and 1650°F. (732°, 815° and 898°C.).

(2) Room-temperature tensile tests on the alloys in the solution-treated and the age-hardened conditions.

(3) Room-temperature tensile tests after 100 hours' exposure at 1500°F. (815°C.), to determine the degree of embrittlement resulting from high-temperature exposure.

The results lead to the following conclusions:

Iron at about 5 per cent. (alloy 5) is apparently quite effective in stabilizing the Ni-Mo structure at about 26 per cent. molybdenum. No data are available on the effect at the 22 per cent. molybdenum level. The nature of the stabilization is uncertain, since the microstructures at 1350° and 1500°F. (732° and 815°C.) indicate that structural changes were taking place in the alloy.

Identical compositional variations, at the 22 per cent. and the 27 per cent. molybdenum levels, indicate that some alloying elements have a greater effect on stability of structure at the higher than at the lower molybdenum content. For example, 1·3 per cent. of niobium has only a slight influence in the 22 per cent. molybdenum alloy, whereas in the 27 per cent. alloy its effect is much more potent. Similarly, alloy No. 1 is stable as compared with alloy No. 4.

It is to be concluded that the changes in structure and properties resulting from alloying additions are due not only to a shift in solvus boundaries, but also partly to the alteration in rate of change of the γ nickel solid solution into the β structure.

The results of the tests confirm the suggestion that the addition of further alloying elements lessens or prevents embrittlement which is found in the binary vacuum-melted nickel-molybdenum alloys.

The authors comment on the influence of temperature on the strength relation between the vacuum-melted alloys and commercial Hastelloy. At 1350°F. (732°C.) alloys 4, 5 and 6 all show better strength than Hastelloy B up to about 500–1,000 hours; at

Alloy	C %	Cr %	Mo %	Fe %	V %	Mn %	S %	Nb %	Ni %
1	0·02	0·65	22·3	0·43	0·38	0·72	0·004	—	bal.
2	0·01	0·64	22·4	4·86	0·39	0·66	0·005	—	bal.
3	0·02	—	22·5	0·70	—	0·74	0·005	1·30	bal.
4	0·02	0·65	28·5	0·16	0·34	0·66	0·005	—	bal.
5	0·02	0·62	26·5	4·80	0·37	0·65	0·006	—	bal.
6	0·03	—	26·5	0·82	—	0·65	0·005	1·29	bal.

1500°F. (815°C.) this superiority is found only in alloys 4 and 6. At 1650°F. (898°C.) Hastelloy B is considerably stronger than any of the modified vacuum-melted alloys, and it is at that temperature that the differences among the vacuum-melted alloys themselves are at a minimum. It is suggested that the relative improvement of the commercial Hastelloy B with rising temperature may be due to its generally higher alloy content, which would include silicon, cobalt and nitrogen as well as the elements which were added to the experimental alloys tested.

High-Temperature Impact Strength of Cermets

H. B. PROBST and H. T. McHENRY: 'A Study of the Impact Behaviour of High-Temperature Materials.' *Nat. Advisory Committee for Aeronautics, Tech. Note* 3894, Mar., 1957; 23 pp.

Report on the evaluation of the impact properties of titanium-carbide-base cermets, some high-temperature alloys, and the intermetallic material NiAl, at room-temperatures and at elevated temperatures up to 1750°F. (954°C.). The N.A.C.A. drop test was employed, and in this connexion a study was made of variables which affect the impact-resistance as measured by that test, e.g., gripping force, gripping material and repeated impact. A modified specimen-supporting arrangement which eliminates the first two of these variables is described.

The impact energies of the cermets were found to vary with the amount of binder present, e.g., a composite containing 50 per cent. of nickel showed a much higher resistance than specimens containing only 10 or 30 per cent. of metal. Earlier investigations have shown that the wettability of the carbide is improved by the addition of molybdenum to the nickel binder, and that a finer carbide-particle size and increased impact-resistance at room temperature is thus achieved. This superiority was confirmed in the present tests, but it was not maintained at 1200°F. (648°C.). A composite in which the binder was an alloy of nickel, cobalt and chromium was, however, characterized by improved strength at both normal and elevated temperature, whereas in nickel-aluminium-molybdenum-bound cermets contradictory results were obtained. It is, therefore, at present impossible to predict the impact strength of a cermet on the basis of the composition of the binder. Decreasing angularity in the particles of the carbide was favourable.

The impact-resistance of cermets was only slightly affected by increase in temperature of test, but the general trend was a fall in strength with rise in temperature.

Comparison of the cermet compositions with representative established and experimental high-temperature alloys of varying composition showed that, on the basis of impact-resistance, most cermets are inferior to alloys which would be classed as brittle.

Titanium-Carbide/Nickel System

See abstract on p. 118.

Grain Growth of Titanium Carbide in Nickel

See abstract on p. 118.

Influence of Ceramic Coatings on Creep Properties

J. R. CUTHILL and W. N. HARRISON: 'Effect of Ceramic Coatings on the Creep Rate of Metallic Single-Crystal and Polycrystalline Specimens.' *Wright Air Development Center, Tech. Report* 56-85; 45 pp. *P.B.* 121,493.

The finding that oxidized surfaces on some metals improved yield and creep properties at room temperature suggested the possible use of coating as a means of securing enhanced creep-resistance at elevated temperatures.

This report is introduced by a summary of the possible mechanisms by which coating layers could influence creep rate. Literature relevant to the respective theories is briefly considered, as a background to a detailed report of experimental work on 80-20 nickel-chromium alloys and nickel, examined in the uncoated and the ceramic-coated conditions. For the alloy specimens various types of ceramic coating were used: on nickel only one type was applied. The tests on the nickel specimens included, in addition to creep tests, determinations of the diffusion of hydrogen through the coated and the uncoated material.

A ceramic coating designated N.B.S. N-143 (barium-silicate type, containing cerium oxide to impart increased refractoriness) was found to improve the creep properties of the 80-20 nickel-chromium alloys at 1975°F. (1080°C.), to a significant extent. The coating almost completely inhibited the formation of voids which were found in uncoated specimens of the alloys. At test temperatures of 1800° and 1900°F. (982° and 1037°C.) coating of these alloys resulted in little or no improvement in creep behaviour under normal rates of strain: at very high strain rates a detrimental effect was observed.

At 1400°F. (760°C.) the diffusion rate of hydrogen through high-purity nickel coated with a ceramic was found to be less than 5 per cent. of the rate of diffusion through uncoated nickel. In creep tests made at that temperature (load 3,650 p.s.i.) there was little difference between the behaviour, in hydrogen, of the uncoated and coated high-purity specimens.

The creep rate, at 1500°F. (815°C.) and 1,000 p.s.i., of ¼-in. single-crystal specimens of uncoated high-purity nickel was found to be unaffected by a change in atmosphere from reducing to oxidizing. The presence of grain boundaries and of carbon was a pre-requisite for the creep rate to be affected by a change in atmosphere from hydrogen to air, and even in that case the change was slight.

A few tests made on 25-20 chromium-nickel stainless steel indicated that coating with ceramic (N.143 type) had a beneficial influence, but the data obtained were insufficient to establish a quantitative difference in the creep behaviour of this group of materials.

M_s Temperatures for Stainless Steels

F. C. MONKMAN, F. B. CUFF and N. J. GRANT: 'Computation of M_s for Stainless Steels.'
Metal Progress, 1957, vol. 71, Apr., pp. 94-6.

A series of investigations has been for some time under way at the Massachusetts Institute of Technology with a view to relating the chemical composition and microstructure of stainless and heat-resisting steels to the high-temperature properties of the materials. In view of the known influence of martensitic ferrite, when formed in such steels, determinations have been made of the M_s temperature of a wide range of laboratory-melted steels. The observations made have been compared with temperatures calculated from an equation proposed by EICHELMAN and HULL (*Trans. Amer. Soc. Metals*, 1953, vol. 45, p. 77). Equations developed as a result of the work at M.I.T. are considered to represent a slightly greater accuracy, but it is believed that 'first-degree formulas relating M_s to chemical composition of the chromium-nickel steels are inherently inaccurate, since the influence of carbon and nitrogen on the martensite point on cooling is quite complex (non-linear) and depends on the whole chemical analysis, in a way as yet unknown'. The authors are, however, of the opinion that such equations should have some practical utility in predicting the M_s temperatures in a wide range of stainless chromium-nickel-iron compositions.

Strain-Hardening in Austenitic Nickel-Chromium Steels

G. W. POWELL, E. R. MARSHALL and W. A. BACKOFEN: 'Strain-Hardening of Austenitic Stainless Steel.'
Trans. Amer. Soc. Metals, 1957, vol. 50, *Preprint* 10: 27 pp. + figures.

In studies of the mechanical behaviour of austenitic stainless steel, the main interest has been concentrated on changes associated with the martensitic transformation of face-centred-cubic austenite (γ) to body-centred-cubic ferritic (α), or low-carbon martensite which may occur in such steels. As a result of the extensive investigations made, it is known that temperature, the stress system, composition of the steel, plastic strain and rate of strain all have a significant influence on the $\gamma \rightarrow \alpha$ transformation. The work has, however, centred mainly on the effect of these variables on elastic behaviour. The aim of the study now reported has been to extend the knowledge of the influence of the respective factors, by investigating their relation to the strain-hardening occurring in austenitic steels, as revealed in stress-strain curves.

The composition of the two steels used is shown in the table below.

Tensile tests, at true-strain rates, and torsion tests, were made at room temperature and at various sub-zero temperatures, and these were supplemented by compression tests at room temperature. The volume fraction of martensite present was determined by density measurements, using the technique described by FLETCHER and COHEN (*ibid.*, 1944, vol. 32, p. 333).

The results of the tests established that the shape of the stress-strain curve is closely dependent on the stability of the austenite. In the absence of any transformation, as, for example, in Type 304 steel deformed at room temperature, the stress-strain curve is of parabolic form. With increasing instability there is marked decrease in the rate of strain-hardening after some plastic strain. In general, the abrupt change in slope (1) occurred at smaller stress as the austenite stability increased, (2) was accompanied by the terminal stage of transformation, and (3) corresponded approximately to the onset of necking in tension.

When tension-tested at $-263°C.$ ($-441°F.$), both steels showed multiple necking and discontinuous extension, with serrated stress-strain curves over the yield-to-fracture region. It is suggested that such instability is due in large measure to localized softening due to heat evolved in testing at a temperature where specific heat is low and flow stress is strongly temperature-dependent.

The effect of rate of strain on the martensitic transformation was found to depend on the magnitude of the rate. At 20°C. increasing the rate of strain from 6×10^{-3} to 60×10^{-3} min.$^{-1}$ stimulated martensite formation, but further increases in rate resulted in less transformation. The cause of the former effect is considered to be the higher levels of stress and strain energy accompanying the higher rate, and the latter phenomenon is regarded as due to a temperature rise in testing.

The mode of deformation is shown to affect the

Composition and Other Characteristics of the Steels

Steel	C	Ni	Cr	Mn	P	Si	S	Lattice Parameters (Ångstrom Units)		A.S.T.M. Grain Size	Measured M_s (°C.)	Vickers Hardness
								Austenite	Martensite			
	%	%	%	%	%	%	%					
301	0·053	7·91	18·11	—	—	—	—	3·590	2·872	6	below −43 above −54	150
304	0·055	10·62	18·05	1·86	0·02	0·52	0·013	3·591	2·872	5 → 6	below −263	160

progress of transformation in the plastic range: uniaxial tension promoted martensite formation more than either compression or torsion. This finding correlates with a comparison of strain-hardening in tension and torsion based on the relationship between maximum shear stress and maximum finite shear strain.

Mechanical Properties of Precipitation-Hardenable Stainless Steels

D. B. ROACH and A. M. HALL: 'The Engineering Properties of Precipitation-Hardenable Stainless Steels.' *Titanium Metallurgical Laby., Battelle Memorial Inst., Report T.M.L.* 48, July 20, 1956; 28 pp. + 4 appendices. *P.B.* 121618.

In order to evaluate the potential usefulness of titanium and its alloys it is necessary to obtain a comprehensive survey of the properties of the steels and alloys most closely competitive. This report comprises a survey of the hardenable stainless steels which have particular interest *vis-à-vis* titanium in the aircraft field.

The review is based on examination of four steels commercially produced in the U.S.A.: Stainless W, 17-7 P.H., 17-4 P.H. and A.M. 350. For compositions see table below.

Two of the steels (17-7 P.H. and A.M. 350) have excellent formability in the annealed condition. All four are relatively simple to fabricate and can be welded and brazed, although to develop high strength in weldments, full heat-treatment is required after welding.

'The steels are not difficult to melt and cast, and could be produced in practically any integrated steel mill having electric-furnace capacity. However, careful control of composition is required to maintain consistent strength properties, and, to ensure typical or guaranteed properties, the heat-treating instructions recommended by producers must be closely followed. Care in handling the steels during fabrication and heat-treatment is also essential: any nitriding or carburization may result in inferior properties, and excessive scaling may also be troublesome.

'Recent improvements which have been made in some of these steels indicate that further increase in their strength properties may be anticipated.'

The present report does not include consideration of specific applications of the precipitation-hardening stainless steels, nor does it contain any direct comparison between the properties of the steels and those of titanium-base materials. Such a comparison is made in companion documents, *T.M.L. Reports* 5A and 13.

Precipitation-Hardening Stainless Steels
(Five Types Developed in the U.S.A.)

	C %	Cr %	Ni %	Ti %	Al %	Cu %	Mo %	N_2 %	Nb+Ta %
Stainless W	0·12	16·0-18·0	6·0-8·0	1·0 max.	1·0 max.	—	—	0·2 max.	—
17-7 P.H.	0·9	16·0-18·0	6·5-7·5	—	0·75-1·50	—	—	—	—
17-4 P.H.	0·07	15·5-17·5	3·0-5·0	—	—	3·0-5·0	—	—	0·25-0·45
A.M. 350	0·10	15·5-17·25	4·0-4·5	—	—	—	2·5-3·0	—	—
A.M. 355	0·17	14·5-16·0	4·0-4·5	—	—	—	2·5-3·0	—	—

In addition, consideration is given to the mechanical properties obtainable in 17-7 P.H. by an improved heat-treatment, and to those of steel A.M.355, an improved version of A.M. 350. All the information presented has been obtained from the producers of the respective steels, and the authors make no critical appraisal of test methods, or of the properties reported. The metallurgical principles involved in heat-treatment of these steels are discussed.

The information assembled is considered to warrant the following conclusions:

'The precipitation-hardening stainless steels offer attractive combinations of properties up to temperatures of 700°F. (370°C.). They have good strength/weight ratios, high moduli of elasticity, good stress-rupture and creep properties, and good fatigue properties. In addition, the steels show similar properties in both the longitudinal and transverse directions, and in tension and compression.

Nickel-Chromium Stainless Steels for High-Temperature Piping

R. M. CURRAN and A. W. RANKIN: 'Austenitic Steels in High-Temperature Steam Piping.' *Amer. Soc. Mechanical Engineers, Paper* 56-A-217, Nov., 1956; 18 pp.

This paper is a further example of literature originating from problems associated with welding of austenitic steel piping. The authors are members of the Steam-Generator Department of General Electric Company, Schenectady, where much research has been carried out on this subject.

To provide a general background upon which research and development programmes can be based, the Company's engineers have examined typical failures by cracking which have occurred in service. This paper presents the case histories of seven representative examples of cracking in, and adjacent to welds in austenitic piping systems. They demonstrate the location of cracking in relation to the weld

deposits and the base materials, the path of cracking within the grain structures, the orientation of the cracking with respect to the axis of the pipe, and the possible sources of the cracking stresses. Good illustrations are shown. Details are given of the plants in which the pipes were, respectively, installed, and of the conditions of operation.

From the observations made, coupled with other extensive experience, it is concluded that the major causes of cracking in the austenitic weldments are notch concentrations, high bending stresses, and residual welding stresses. The difficulties encountered can be substantially minimized by:

(1) avoidance of notches or sharp changes in section, especially in the vicinity of welded joints,

(2) ensuring that the piping systems move in accordance with design, to prevent the imposition of excessively high bending stresses,

(3) providing correct post-welding heat-treatment of all joints.

Still further improvement is hoped for as a result of the development of the 16-8-2 chromium-nickel-molybdenum-iron electrode (see also p. 138 of this Bulletin) and from information obtained by means of the hot-ductility test (see also paper on p. 137).

Austenitic Chromium-Nickel Steel Piping: Specification

AMER. SOC. TESTING MATERIALS: 'Tentative Specifications for Welded Large-Outside-Diameter Light-Wall Austenitic Chromium-Nickel Alloy Steel Pipe for Corrosive or High-Temperature Service', contained in Report of A.S.T.M. Committee A-1 on Steel.

A.S.T.M. Preprint 1, June 1957, pp. 21-6.

Eight grades of steel are covered, containing, in various proportions, nickel 8-15, chromium 16-26, per cent. They are representative of A.I.S.I. Types 304, 309, 310, 321, 347, 316, 317 and 348. The sizes of piping to which the specification refers are 14-30 in. inclusive in nominal diameter, with extra-light and light wall thicknesses.

Process and fabrication requirements cover production, heat-treatment and welding. Tensile properties called for are U.T.S. 75,000 p.s.i. min. (33·5 tons per sq. in.); yield strength 30,000 p.s.i. min. (13·5 tons per sq. in.). Elongation in 2 in. is to be 30 per cent. min. Bend and pressure tests are also incorporated in the specification.

High-Temperature Brazing of Heat-Resisting Alloys

W. E. RUSSELL and J. P. WISNER: 'An Investigation of High-Temperature Vacuum and Hydrogen Furnace Brazing.'

Nat. Advisory Committee for Aeronautics, Tech. Note 3932, Mar., 1957; 29 pp.

The use of light-weight sheet-metal components for turbo-jet engines, which is now being considerably extended, depends to a large extent on successful furnace brazing, and the technique is therefore currently under intensive investigation.

In work at the Lewis laboratory of the National Advisory Committee for Aeronautics little difficulty was encountered in obtaining adequate joint coverage and satisfactory shear strength in brazed joints, but mechanical tests and metallographic examination showed that the brazing operation had damaged the base metal, resulting in loss of strength and impaired ductility.

With a view to development of materials and procedure which would give improved properties, an investigation was carried out to study the influence of various temperatures and of time at the brazing temperature, on the tensile strength of sheet specimens coated with braze, and on the shear strength of brazed joints. A further object of the research was to develop methods of minimizing base-metal damage, while still providing adequate joint coverage and strength.

The compositions of the basis metals and of the brazing alloys used are tabulated (see p. 136). Nicrobraz was employed as typical of the boron-containing type of brazing alloy, while the type in which silicon is used was represented by the G.E.C. 81 material.

For plating of the basis metals both electroless nickel and electrodeposited nickel were used. The vacuum and hydrogen furnaces employed are described, together with details of brazing technique and testing and evaluation procedure used. The temperatures used in brazing were 2100° and 2150°F. (1148° and 1176°C.) and the mechanical tests were made at 1200°F. (648°C.).

In general, the results show that the boron-free type of alloy is less damaging to the base metal than is the boron-bearing type, but the shear strength of the joints made with the boron-containing alloy was the better of the two. It is suggested that the higher carbon content of the Nicrobraz alloy may be significant. The temperature of furnace brazing and the time at temperature were important factors in relation to the strength and elongation of braze-coated alloys: these effects varied according to the basis metal and the brazing medium used.

Shear specimens of all four base alloys brazed in hydrogen, with both types of brazing alloy, exhibited erratic joint coverage by the brazing alloys. If, however, joint coverage was complete, vacuum and hydrogen brazing produced joint shear strengths of about the same magnitude.

Alloys of the nickel-base type containing titanium and aluminium could be successfully brazed in vacuum by first electroplating with nickel, but for hydrogen brazing of such alloys addition of flux to the brazing alloy proved more reliable than plating. Flux additions were found to be ineffective during vacuum brazing.

On the nickel-base age-hardening type of alloy electroless nickel plating enhanced flow and joint coverage, but it was found to have an adverse effect on the strength and ductility of the base metal and on the shear strength of the joints.

Brazing of High-Temperature Materials with Nickel-Chromium-Boron Alloy

K. A. PEEL: 'Nicrobraze Applied to the Napier 'Eland' Gas Turbine.'
Welding and Metal Fabrication, 1957, vol. 25, Apr., pp. 137–43.

The article contains a report of tests carried out on butt-joint specimens of R.ex 337 (complex austenitic chromium-nickel steel) and on Tee joints of Nimonic 75 (nickel-chromium-base alloy) and Stellite X 40 (cobalt-base alloy containing nickel, chromium and tungsten). The brazing alloy used was Nicrobraze, of which the nominal composition is given as nickel 65–75, chromium 13–20, boron 2·75–4·75, per cent.

Factors which are of primary importance in the successful brazing of high-temperature alloys are reviewed; particular attention is directed to the need for suitable furnaces and for control of atmospheres. The application of Nicrobraze is also described as demonstrated by its use in assembly of the hollow nozzle blades for the three stages of the Napier 'Eland' turbo-prop engine.

Resistance of Brazing Alloys to Attack by Sodium and to Oxidation.

G. M. SLAUGHTER, C. F. LEITTEN, P. PATRIARCA, E. E. HOFFMAN and W. D. MANLY: 'Sodium Corrosion and Oxidation Resistance of High-Temperature Brazing Alloys.'
Welding Jnl., 1957, vol. 36, May, pp. 217S–25S.

The work reported was carried out in the Oak Ridge National Laboratory, Oak Ridge, Tenn.

It is pointed out that in addition to the problems normally associated with the operation of heat-exchanger equipment and other high-temperature plant, the use of liquid-metal coolants and of other corrosive media at high temperatures has complicated the selection of structural materials for many applications. It is obviously essential that materials used in joining the high-temperature components should also be resistant to attack by the various corrodents encountered in service.

Although welding is extensively employed in construction of high-temperature equipment, brazing is particularly suitable for some applications, and a long-range development and evaluation programme has been under way for some years, with the object of obtaining fundamental information on the feasibility of using brazing alloys in high-temperature heat-exchangers. Many commercial brazing alloys have been examined, in comparison with experimental types developed at the Oak Ridge and other laboratories. A list of the alloys which have been tested is given in this report, and the methods used in evaluation are outlined. These include metallurgical examination, physical and mechanical tests, determination of flow qualities and ductility, and corrosion and oxidation tests.

This paper is concerned solely with the resistance of brazing alloys to corrosion by liquid sodium and to oxidation: reference is made to other characteristics of the alloys only where such properties are pertinent to their corrosion- and oxidation-resistance.

In discussing the nature of the alloys selected for test, the authors point out the peculiar suitability of nickel-base systems for use as brazing alloys: a very large proportion of the alloys examined were nickel-base types.

The equipment and technique used for the dry-hydrogen brazing (of nickel, stainless steels and Inconel) are described and illustrated. Tests of the brazed joints in liquid sodium were made under static conditions involving exposure for 100 hours at 1500°F. (815°C.) and by a 'see-saw' method, in which sodium was circulated for 100 hours at a hot-zone temperature of 1500°F. (815°C.). In the latter tests the temperature differential was approximately 400°F. (222°C.). The static test was used mainly for rapid screening of the alloys: the see-saw procedure constituted a dynamic test, in which movement of the corrodent was secured by oscillation of the furnace. (The paper includes an illustration of the see-saw test furnace, and the loading procedure is diagrammatically shown.)

In the oxidation experiments the specimens were tested both statically and with intermittent cycling to room temperature, in order to simulate service conditions which might give rise to spalling of oxide. Test temperatures were 1500° and 1700°F. (815° and 926°C.), and the influence of time at temperature was studied. The furnace used was a resistance-wound box type, having a hot zone approximately $5 \times 8 \times 14$ in. No circulation of air was maintained in the furnace other than that arising from natural convection.

The results of the respective tests are presented in a comprehensive series of tables, and the condition of typical joints after corrosion and oxidation exposure is illustrated by photomicrographs. The extensive experimental data obtained lead to the following evaluation of the alloy types examined:

'A wide variety of brazing alloys possess adequate resistance to sodium or to oxidation, and several alloys exhibited good resistance to attack by both media. The most promising are listed on page 137.

'The nickel-silicon-boron-iron alloy (No. 1) was one of the alloys which exhibited resistance to both corrosion and oxidation. Alloys Nos. 2, 3, 6 and 7 are other boron-containing alloys possessing good compatibility in both environments.

'Alloys of the nickel-chromium-silicon type also possess good corrosion- and oxidation-resistance at 1500°F. (815°C.).

'The 80-10-10 nickel-chromium-phosphorus alloy was shown to possess fair resistance to attack. An undesirable characteristic of some phosphorus-containing alloys should, however, be mentioned. Experiments have indicated that extreme brittleness may be present, owing to the formation of complex intermediate phases. This condition was evident in an 88-12 nickel-phosphorus alloy.

'No precious-metal alloys were tested in sodium, since earlier research had indicated that a definite incompatibility existed at the elevated temperatures under consideration.

No.	Ni	Si	B	Fe	Cr	P	C
1	91·2	4·5	2·9	1·4	—	—	—
2	82·1	4·5	2·9	3·5	7·0	—	—
3	83·4	5·0	3·0	2·5	6·0	—	0·1
4	70	10	—	—	20	—	—
5	80	—	—	—	10	10	—
6	93·2	3·5	1·9	1·4	—	—	—
7	73·2	4·5	3·5	4·5	13·5	—	0·8

'Although most of the alloys tested showed a high degree of oxidation-resistance, a few, such as the 60–40 manganese-nickel alloy, were severely attacked.

'In evaluating the corrosion- and oxidation-resistance of a brazing alloy, emphasis should be placed on the results of see-saw and cyclic tests, since these conditions more nearly simulate the expected operating conditions than do static tests.'

Hot-Ductility Testing of High-Temperature Steels and Alloys

E. F. NIPPES, W. F. SAVAGE and G. GROTKE: 'Further Studies of the Hot Ductility of High-Temperature Alloys.'
Welding Research Council Bull. No. 33, Feb., 1957; 32 pp.

This Bulletin contains a detailed report of the progress, to March 1956, in the investigations on hot ductility of high-temperature materials which is being carried out in the Rensselaer Polytechnic Institute. The research is directed specifically to establishing a relationship between hot ductility and the quality and serviceability of high-temperature materials, particularly in the welded condition.

In general, welding problems in this field fall into one of three categories: (1) impairment of corrosion-resistance, (2) impairment of mechanical properties, and (3) cracking in the weld or in the weld-heat-affected zone. Of these, cracking is least well understood, and conflicting ideas have been expressed as to the cause, origin and nature of the cracking. Qualitative examination of typical cracks has, however, resulted in classification into seven types: crater, star, micro, fusion-line, root, base-metal and stress-relief cracks. All types are produced as a result of the combined action of high shrinkage stresses and triaxiality of stress. Data supporting this statement are presented in the report.

Consideration of available data (including the significance of the liquation of low-melting boundary films or other foreign phases at the grain boundaries) led to the planning of a research with the aim of:

(*a*) developing testing equipment capable of evaluating the effect on the elevated-temperature properties of alloys, of thermal cycles involved in welding;

(*b*) utilizing such equipment for studying the effect of thermal cycles on typical high-temperature alloys;

(*c*) correlating the results of such tests with the weldability of the alloys.

A detailed and well illustrated description is given of the hot-ductility testing equipment used (known as 'Gleebel'). It provided, *inter alia*, for a maximum reproducible heating rate of 3000°F./sec. (1667°C./sec.); a maximum reproducible cooling rate of 350°F./sec. (195°C./sec.), and a maximum practical specimen temperature of 2500°F. (1370°C.). Operating technique is fully explained.

The test materials used were representative of 34 different types; some specimens were from structural parts of steam turbines, and some from high-temperature steam piping, some were supplied by producers, and others were from warehouse stock. Detailed analyses of the respective types are given in the report. Particular attention was devoted to Type 347 austenitic chromium-nickel steel, first, as being much used in welded construction for high-temperature plant and second, as a material in which weld cracking has been a major problem.

The results of the tests are shown as (1) per cent. reduction in area, (2) total strain at fracture, and (3) ultimate tensile strength, all three values being recorded as functions of the test temperature. The results are reported in a series of more than 50 graphs.

Most of the materials, when tested at the peak temperature of the thermal cycle involved, exhibited properties different from those found in tests at the same temperature during the cooling portion of cycles having a peak temperature near the melting point of the alloy. Two major classifications of behaviour were therefore made, to differentiate between 'tested-on-heating' and 'tested-on-cooling' behaviour.

As a result of the observations made, two mechanisms are proposed as possible explanations of cracking phenomena; the information at present available is considered to be inadequate to allow of defining the exact nature of the reaction responsible. One explanation is based on a grain-boundary precipitation reaction, the other on a mechanism involving incipient melting at the grain boundaries. The two theories are critically discussed.

Individual sections of the report deal specifically with the behaviour characteristic of various groups of material, e.g., Type 347 chromium-nickel steels,

other austenitic steels of the 300 series, N-155 and other miscellaneous proprietary alloys, and nickel-base alloys typified by Hastelloys B and C, Inconel X, Inconel, and a nickel-zirconium alloy.

The test results, which demonstrated that hot-ductility values correlated well with service behaviour wherever the latter was known, are considered to warrant the conclusion that the hot-ductility test provides a sensitive means of evaluating the effect of rapid thermal cycles on the performance of structural materials up to temperatures of 2500°F. (1370°C.).

The following general conclusions are drawn:

'Two general types of elevated-temperature behaviour were observed when materials were tested for hot ductility on heating.

(*a*) Most materials showed a gradual increase in ductility with rise in test temperature, up to a temperature slightly below the melting range. At that critical stage the materials failed in a completely brittle manner. This behaviour is classed as H.1.

(*b*) A few materials showed a gradual loss of ductility as the test temperature was raised above some intermediate range. Such behaviour is classed as H.2.

'When the materials were exposed to thermal cycles involved in welding, and tested at various temperatures on cooling, three types of behaviour were observed:

(1) When the tested-on-cooling behaviour was the same as that on heating the material is classed as exhibiting C(....)1 behaviour. (The points within the brackets represent the relevant peak temperature which must be inserted for evaluation of any individual material.)

(2) When the tested-on-cooling behaviour was essentially the same as the tested-on-heating behaviour above 2100°F. (1248°C.), but showed less ductility in the range 1800°-2000°F. (982°-1092°C.), the material was given the designation C(....)2.

(3) When the tested-on-cooling ductility was significantly below that found for testing-on-heating the designation C(....)3 was allotted.'

'In general, materials exhibiting either Class H.2 or Class C(....)3 behaviour are believed to be prone to cracking, either during welding or in subsequent service.

'No definite correlation has yet been possible between composition and behaviour in the hot-ductility test.'

see also

Hot Ductility of Austenitic Steels in Relation to Quality of Welds

H. M. SOLDAN and C. R. MAYNE: 'Ductility related to Service Performance of Heavy-Wall Austenitic Pipe Welds.'
Welding Jnl., 1957, vol. 36, Mar., pp. 141S-7S.
Reprint issued by INTERNATIONAL NICKEL CO., INC.

The purpose of this paper is to show the important part which ductility plays in the fabrication and performance of welds in heavy-wall austenitic steel piping, with particular reference to service in high-temperature central stations. The report is closely associated with other papers which have been published in this journal emphasizing the significance of hot ductility as a factor determining the behaviour of austenitic welds at elevated temperatures: see, for example, a paper abstracted in *Nickel Bulletin* 1957, vol. 30, No. 6, pp. 102-3. Observations of the service behaviour of niobium-stabilized austenitic nickel-chromium steel piping in high-temperature power stations has correlated closely with the results of laboratory tests, and it may be confidently anticipated that this approach to the problem will materially assist in setting up specifications for piping materials which will give reliable life over long periods.

Attention is also directed to the fact that welds in highly restrained thick-wall piping, which have been difficult to make on account of the poor hot ductility of the base metal have been successfully achieved by using a new type of electrode, which contains chromium 16, nickel 8, molybdenum 2, per cent. The improvement obtained by the use of these electrodes is attributed to the relief of residual welding stresses resulting from the better ductility of the weld metal deposited.

Brazing of Copper-Nickel Alloys

See abstract on p. 119.

Nickel and Nickel-Clad Materials for Electron Tubes

See abstract on p. 108.

Welding of Stainless-Clad Steel in Nuclear Reactors

W. LEONARD and J. C. THOMPSON: 'Peripheral Welding of Internally-Clad Steel for Nuclear Reactor Application.'
Welding Jnl., 1957, vol. 36, Mar., pp. 243-51.

In many designs of interior-clad pressure vessels or piping access for welding is from one side only, the outer surface. Since back chipping is not possible, an alternate welding procedure must be developed. This is the type of problem with which the present paper is concerned, as exemplified by welds made in stainless-steel-clad vessels.

Two basic types of joint were studied, with variations in design and filler metals. The special welding technique and inspection procedures used to secure the high quality essential in joints incorporated in nuclear reactors are described, with many illustrations. The results of the tests made on welds prepared by various methods confirm that stainless-clad steel having access for welding from the carbon-steel surface only may be satisfactorily welded by using correct joint design and welding technique.

The use of an ingot-iron insert to eliminate the problem of dilution of the stainless-steel/carbon-steel material introduces a notch effect into the joint, and it was found that this notching caused failure in practically all of the bend-test specimens used in the

research reported. The following recommendations are made with regard to effecting the transition from stainless-steel weld metal to carbon-steel weld metal:

(*a*) Low-carbon stainless-steel filler metal should be used to overlay the austenitic weld metal, and should be deposited to a height of at least $\frac{3}{16}$ in. above the stainless-steel/carbon-steel interface. An intermediate layer of ingot-iron filler metal may overlay the low-carbon stainless-steel filler metal. The minimum thickness of this layer should be $\frac{3}{16}$ in., in order to act as an effective barrier.

(*b*) Elimination of the intermediate ingot-iron weld-metal layer is possible if low-hydrogen weld metal is deposited on an extra-low-carbon 19-9 stainless steel. All possible precautions should be taken to ensure minimum dilution at the stainless-steel/carbon-steel interface.

Although martensite and other transition phases are present at the interface of the carbon steel and the stainless steel, it is to be concluded that these transition products, having a low carbon content, do not seriously affect the ductility of the welded joint.

Influence of Surface Condition on Nickel-bonded Titanium Carbide

L. ROBINS and E. M. GRALA: 'Preliminary Investigation of the Effect of Surface Treatment on the Strength of a Titanium Carbide-30 per cent. Nickel-base Cermet.'

Nat. Advisory Committee for Aeronautics, Tech. Note 3927, Feb., 1957; 16 pp.

Since cermets are relatively brittle, small surface imperfections may be expected to result in a large loss of strength and impact-resistance, with consequent effect on reliability in service. Such surface imperfections may be caused by fabrication operations, or may result from service conditions (e.g., erosion, abrasion, corrosion and oxidation).

Study of the influence of surface condition has been made by investigation of the room-temperature modulus of rupture and impact strengths of titanium carbide/nickel-base cermet specimens which had undergone various surface treatments (grinding, lapping, blast cleaning, acid roughening and oxidizing). Several of the treatments were selected as simulating conditions obtaining in production and/or operation of cermet turbine blades: others were intentionally exaggerated, to represent a poorly ground surface, steel-grit blasting, etc.

The composition of the cermet used (K152B) was nominally (by weight) 62 per cent. of titanium carbide, 8 per cent. of a complex carbide of niobium, tantalum and titanium, and 30 per cent. of nickel. The structure consisted of a mixture of angular carbide particles, metal binder (a nickel-rich solid solution) and possibly some graphite. Representative microstructures are illustrated. The nature of the tests and the conclusions drawn from the results are summarized as follows:

'The average strengths after the various treatments varied from about 200,000 to 80,000 p.s.i.* in modulus of rupture and from about 3·5 in. to 1 in.-lb. in impact-resistance. The strengths of lapped, grit-blasted, diamond-ground or vapour-blasted specimens did not vary significantly. The most serious loss of strength occurred after oxidation (at 1600°F.: 870°C.) for 100 hours, surface roughening by acid attack, and severe grinding with a 60-grit silicon-carbide abrasive wheel. The modulus-of-rupture value of oxidized specimens was improved by subsequent grit-blasting or re-grinding with a diamond abrasive wheel.'

High-Temperature Hardness Testing

M. SEMCHYSHEN and C. S. TORGERSON: 'Apparatus for Determining the Hardness of Metals at Temperatures up to 3000°F. Elevated-Temperature Hardness of Iron, Molybdenum, Tungsten and S-816.'

Trans. Amer. Soc. Metals, 1957, vol. 50, *Preprint* 12; 7 pp. + table and figure.

The apparatus described is illustrated by a line drawing. The principles of the pyramid penetration method of hardness testing are embodied in it, using a synthetic sapphire as the penetrator medium. The hardness determinations are made in an atmosphere of purified argon.

The authors report hardness determinations on pure iron and on S-816† alloy from room temperature to 2300°F. (1260°C.) and values for molybdenum and tungsten up to 3000°F. (1649°C.).

Scaling of 18-8 Chromium-Nickel Steels

J. O. EDSTROM: 'Scaling of 18-8 Stainless Steel in Re-heating Furnace Atmospheres.'

Jnl. Iron and Steel Inst., 1957, vol. 185, Apr., pp. 450-66.

This paper reports work carried out at the Swedish Institute for Metal Research, Stockholm, under the aegis of Jernkontoret. Most re-heating furnaces in Sweden are oil-fired, and many of them employ heavy-oil fuels, which are comparatively cheap. Liability to surface defects on steel has increased with wider use of the heavy sulphur-rich oils, especially in the heating of sheet and slab, on which from time to time flaky and brittle oxide films are formed. On subsequent rolling such films give rise to surface defects. In other cases the oxide films formed are tough and adherent and are less liable to cause defects during processing.

The main object of this investigation was to define the conditions under which oxide films become either flaky or adherent, and to make a study of the structure of the respective types of film, and the mechanism of their formation. The paper also includes an explanation of the varying influence of sulphur on the oxidation process, as affected by the oxidizing or reducing nature of the atmosphere.

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* 89·5 to 35·75 tons per sq. in.

† Complex cobalt-nickel-chromium alloy containing molybdenum, tungsten and niobium.

As a background to the discussion of the experimental results, the author describes the thermodynamics and kinetics of the reactions involved in oxide formation and discusses the nature of the oxides which can form on a steel of the 18–8 chromium-nickel type. The steels used for the experimental work were to the following specification:

Designation	Specification Composition						
	C %	Si %	Mn %	P % max.	S % max.	Cr %	Ni %
S1	0·06	0·5	0·4	0·030	0·030	19	10
S2	0·06	0·5	0·4	0·030	0·030	19	10
S3	0·10	0·5	1·0	0·030	0·030	17·5	10
S5	0·06	0·05	0·4	0·030	0·030	19	10

Steel S2 was ground on No. O emery paper and washed in acetone before being oxidized. Steels S1, S3 and S5 were supplied in the bright-annealed condition and oxidized without any further surface treatment other than successive washing in benzene, soapy water, water and acetone. In all the experiments an oxidation temperature of 1050°C. was used and the rate of gas flow was about 5 cm./s. A test was also made in humid air at 1000°C. A drawing shows the apparatus used for the oxidation experiments.

The compositions of the various oxidizing atmospheres and the time and temperature of exposure are tabulated for the twenty variant conditions used, and the type of oxide formed in each of these conditions is shown. Numerous photomicrographs illustrate the appearance of the surfaces after exposure.

The findings are summarized below:

Sulphur-Free Gas

'Reducing' Atmosphere (CO excess)	'Oxidizing' Atmosphere (O_2 excess)
Thin adherent oxide Tendency to carburization	Thin adherent oxide after short oxidation Flaky, thick oxide film after long oxidation

Sulphur-Containing Gas

Moderately 'Reducing' Atmosphere	Heavily 'Reducing' Atmosphere (CO 10%)	'Oxidizing' Atmosphere
Adherent thick oxide film Sulphide formation Tendency to carburization	Thin adherent oxide Traces of sulphides Carburization	As for sulphur-free gas — —

Flaky oxide films formed in various ways consist principally of iron oxides. In an 'oxidizing' atmosphere a transition takes place from adherent to flaky oxide, which is associated with a chemical breakdown of an early-formed, slowly-growing oxide film of high chromium content. The phase sequence varies according to whether the atmosphere is oxidizing or reducing.

The author proposes a hypothesis for the mechanism of oxide formation on 18–8 chromium-nickel steel, providing an explanation of the structural differences found in scales formed under dissimilar oxidation conditions. In 'oxidizing' atmospheres the presence of sulphur has no effect on the oxidation result, whereas in a 'reducing' atmosphere the presence of sulphur causes sulphide formation and a substantial rise in the rate of scaling. In 'heavily reducing' atmospheres, however, the oxidation-promoting effect of the sulphur disappears.

The differing influence of sulphur on the oxidation mechanism in oxidizing and in reducing atmospheres is explained by calculations of the sulphur vapour pressure in different gaseous mixtures. In a mixture produced by combustion of an oil under conditions of air deficiency sulphur vapour pressures are high enough to permit the formation of low-melting-point sulphides of nickel, iron, etc. Under conditions of about stoichiometric combustion of the oil, or combustion in excess of air, the sulphur vapour pressures are too low for sulphides to form.

The system C–H–O–S at 1050°C. is illustrated in a logarithmic diagram, from which it is possible to read off the partial pressures of the gaseous constituents for every oxygen pressure.

Resistance of Aluminium-Coated Steel to Corrosion by Fuel Ash

J. E. SRAWLEY: 'Aluminum Coatings on Stainless Steel.'

U.S. Naval Research Laby., Washington, D.C.; *N.R.L. Report* 4838, Oct. 17, 1956; 39 pp.

Study of the resistance of aluminium coatings on stainless steel, to attack by residual fuel-oil ashes, has been sponsored by the U.S. Naval authorities as part of the broad programme of work on corrosive attack on structural components of boilers and gas turbines.

In the investigation now reported a stainless steel

of the 25-20 chromium-nickel type was dip-coated with aluminium and exposed to corrodent ashes containing varying amounts of vanadium pentoxide and other constituents. Method of coating, and subsequent heat-treatment of the specimens, conditions of exposure, and inspection procedure are fully described. The tests were made at a temperature of 1700°F. (926°C.).

Within the limitations of the experimental procedure used, aluminium-coated stainless steel is not superior to the uncoated steel in resistance to attack, and to the date of the report no direct reference to aluminium coatings for protection against fuel-ash attack had been found in the literature. The author urges the limitations of rapid screening tests as a means of assessing behaviour under the complex conditions obtaining in boilers and turbines fired with residual fuels.

The report includes a particularly full series of photomicrographs showing the condition of specimens of uncoated and aluminium-coated steels after varying periods of exposure.

Corrosion by Fused Sodium Hydroxide

E. M. SIMONS, N. E. MILLER, J. H. STANG and C. V. WEAVER: 'Corrosion and Components Studies on Systems containing Fused NaOH.'
Battelle Memorial Inst., Report B.M.I. 1118, July 30, 1956; 44 pp.

Work carried out to date to determine the potential value of various hydroxides as coolants or moderators for high-power-density reactors has shown that, at temperatures of 1100°F. (590°C.) and above, all possible container materials, when exposed to fused caustics, are attacked to a degree which would preclude their use in large-scale engineering systems. It appears possible, however, that a long-life reactor having a lower peak temperature is possible. In this connexion, the programme of research on which this investigation was based covered specifically the following corrosion problems:

(1) The rates at which nickel is corroded and 'mass-transferred' by high-temperature NaOH (900°-1500°F.: 482°-815°C.) circulated in small, polythermal flow-loop systems.
(2) The effectiveness of some 60 additive substances in inhibiting the corrosiveness of NaOH to nickel at the higher temperatures.
(3) The resistance of several materials* to attack by NaOH, as determined in conventional isothermal capsule experiments.

In all natural- and forced-convection loops of nickel in which hydrogen-blanketed NaOH was circulated at peak temperatures of 1200°-1500°F. (648°-815°C.) with temperature cycles in the range 100°-200°F. (56°-112°C.) the cold zones plugged severely by nickel deposition in times ranging from less than 10 hours to 200 hours. Considerably better results were, however, obtained in experiments with all-nickel pumping systems in which the peak temperatures were 900° and 1000°F.(482° and 538°C.).

* Graphite of various types, beryllia, beryllium-copper, Borolite (ZrB_2), mild steel and Zircaloy.

The results of this series indicated that in the lower temperature range a reasonable life is possible for a nickel flow system handling NaOH, although a relatively low fluid-temperature cycle would be required.

None of the inhibitors tested effectively suppressed the corrosiveness of NaOH at 1500°F. (815°C.). Only MgO had a noticeable influence, and even with this medium the improvement was not such as to eliminate the problem.

In static tests various grades of commercial graphite were resistant to NaOH at 705°C., but were attacked at 760° and 815°C.

The final section of the report discusses general engineering requirements for components of large-scale flow systems of the type under consideration.

Corrosion by Fuming Nitric Acid: Inhibition by Hydrofluoric Acid

C. E. LEVOE, D. M. MASON and J. B. RITTENHOUSE: 'Inhibiting Effect of Hydrofluoric Acid in Fuming Nitric Acid, on Corrosion of Austenitic Chromium-Nickel Steels.'
Corrosion, 1957, vol. 13, May, pp. 321t-8t.

Previous investigations had shown that with aluminium alloys and certain steels (including an austenitic chromium-nickel type) the presence of 0·2-1·0 wt. per cent. of hydrofluoric acid in fuming nitric acid markedly inhibited corrosion. This later report covers a detailed study of the inhibiting action, as exemplified by tests on 19-9DL and 19-9DX (chromium-nickel steels containing molybdenum and tungsten) and on a welded titanium-stabilized 18-8 chromium-nickel steel. The tests comprised exposure to fuming nitric acid in both the liquid and the gaseous phases, with and without addition of hydrofluoric acid, under conditions pertinent to those obtaining in rocket-propellant service.

The effect of the condition of the stainless steel was investigated by using two sets of specimens, one of which had been annealed at 1800°F. (982°C.) and air-cooled, while the other had been work-hardened and subsequently stress-relieved at 1200°F. (650°C.). In general, specimens which had undergone the work-hardening+stress-relief treatment showed lower resistance to corrosion than the annealed samples.

It was found that the presence of about 0·6 per cent. of hydrofluoric acid, in thermally stable fuming nitric acid containing about 13 wt. per cent. of NO_2 and 3 wt. per cent. of H_2O, markedly inhibited corrosive attack on the materials examined, in all the conditions in which they were exposed. It is, however, important that the steel should not previously have undergone pitting corrosion. On specimens exposed in the corroded condition attack by fuming nitric acid was aggravated by the addition of hydrofluoric acid.

With the proviso of being in the uncorroded condition, the austenitic steels examined are suitable for use in contact with HF-inhibited fuming nitric acid (liquid or gaseous) for periods of at least one

month, at a steady temperature as high as 160°F. (70°C.) or with a temperature fluctuating between 70° and 160°F. (20° and 70°C.).

Resistance of Stainless Steels to Corrosion by Geothermal Steam

T. MARSHALL and A. J. HUGILL: 'Corrosion by Low-Pressure Geothermal Steam.'
Corrosion, 1957, vol. 13, May, pp. 329t-37t; disc., p. 337t.

Experiments to assess the potentialities of geothermal heat for commercial applications are under active prosecution by the New Zealand Government, with particular reference to possible use in electric-power production. Estimates made in 1955, based on utilization of the steam in turbo-generators, indicate a potential available power output, from bores already made in the Wairakei area, of 40,000 kw.

Preliminary examination of equipment used at Wairakei, and experience with boreholes at Rotorua, indicated that the geothermal steam would be more corrosive than normal boiler-plant steam, and in the absence of reliable published information on the subject, tests were carried out to provide the data which would be required by engineers designing the large-scale plant. Fluid representative of that found in the bores at Wairakei was used in the tests, and the conditions were varied to cover those which might be expected to occur in various parts of the plant. The variables studied were the nature of the corrosive medium, time, temperature, and velocity, thus providing for both corrosive and erosive conditions of varying severity.

A wide range of materials was tested, see list below. With the exception of titanium, all were commercial stock typical of materials commonly used in steam plant.

Ferrous Materials

Carbon steel; low-alloy manganese, molybdenum, manganese-molybdenum, chromium-molybdenum, and nickel steels; 13 per cent. chromium steel; seven types of austenitic chromium-nickel steel of simple and complex composition; grey cast iron, high-silicon cast iron, and high-alloy (Ni-Resist) cast iron.

Non-Ferrous Materials

Copper, three bronzes, brass (60-40 type); Monel (nickel-copper alloy); Inconel (nickel-chromium-iron alloy); nickel; aluminium; titanium; nickel-plated, chromium - plated, and galvanized steel.

The results confirm that the geothermal steam is more corrosive than clean boiler-plant steam, due to the impurities present (particularly chlorides, hydrogen sulphide and carbon dioxide). Under the test conditions used, most of the alloys proved moderately susceptible to corrosion. Titanium and the chromium-nickel stainless steels (especially the molybdenum-bearing type) were, however, highly resistant. In the presence of air or oxygen in the geothermal fluids the attack was severe, indicating that in any plant utilizing geothermal steam it will be necessary to avoid standby corrosion or accidental ingress of air.

Sulphide stress-corrosion is a potentially serious hazard in handling geothermal steam, particularly in turbines. In the laboratory tests, stress-corrosion cracking occurred in hardened 13 per cent. chromium steel, hardened low-alloy steels, a straight 18-8 chromium-nickel steel, and aluminium bronze. Hydrogen embrittlement is a further factor requiring consideration, especially in materials used for equipment handling geothermal fluids at high pressure.

Nickel-containing Materials in Plant for Production of Phosphoric Acid

C. C. LEGAL, J. N. PRYOR, T. O. TONGUE and P. L. VELTMAN: 'Phosphoric Acid by the Clinker Process.'
Industrial and Engineering Chemistry, 1957, vol. 49, Mar., pp. 334-7.

All the wet phosphoric acid produced in the U.S.A., and most of that made in other parts of the world, is manufactured by a process producing a relatively weak acid. Strong acid is then obtained by concentration. The present paper describes a procedure by which it is possible to obtain strong wet phosphoric acid without concentration.

One section of the paper deals with tests on materials of construction for use in contact with phosphoric acid. The following conclusions are drawn:

> 'Lead and Type 316 (molybdenum-containing stainless steel) gave the best results for use in leach tanks and filters, and the same steel gave the best results of any materials that are regularly made into filter cloth. Aloyco 20 or Carpenter 20 stainless steels (copper- and molybdenum-containing nickel-chromium steels) and Teflon were satisfactory, but because of the expense and the limited forms in which they are manufactured, these materials are restricted in their use. Mild steel gave satisfactory service for the mixer shell. Nickel and Monel proved unsuitable in the conditions involved. A suitable material for the mixer blades remains a problem.'

Resistance to Hydrogen Fluoride and Hydrofluoric Acid: Review

E. LINGNAU: 'The Behaviour of Materials in Contact with Hydrogen Fluoride and Hydrofluoric Acid.'
Werkstoffe und Korrosion, 1957, vol. 8, Apr., pp. 216-33.

Comprehensive review of information, supplementing that published in 1950 by E. FRANKE (*ibid.*, 1950, vol. 1, pp. 35-9). The information presented, which is supported by a *bibliography* of 201 items, is dealt with in sections relating, respectively, to the production, processing and use of hydrogen fluoride and hydrofluoric acid, and the behaviour of metallic and non-metallic materials of various types in contact with these corrosives. The metals and alloys considered include nickel and nickel-base alloys.

Corrosion-Resisting Applications of Monel and Nickel

'Paper, Gasoline and Fertilizer: Three Cases for Nickel.'

Industrial and Engineering Chemistry, 1957, vol. 49, Apr., pp. 75A–6A.

Notes on three applications in which the resistance of nickel and its alloys to corrosion has increased efficiency and maintained unbroken operation.

(1) *Suction-press rolls employed in paper-making mills* are traditionally made from bronze. Although this material is generally satisfactory, frequent shutdowns are required for reaming and surface re-finishing, due to the build-up of fibres and chemicals in the holes through which the extracted liquid passes. The bronze rolls also corrode, and the resulting roughened surface causes excessive wear of the felt which is on the under side of the paper sheet. Various methods have been tried to alleviate these troubles, but have proved only partially successful. It is now reported, however, that the difficulties have been overcome by the use of Monel for the suction roll. The following service report is made by Union Bag-Camp Paper Company, Savannah, Ga., U.S.A.:

'As a result of the successful use of Monel suction-press rolls on smaller paper machines in another mill, a roll 144 in. long by 36 in. in diameter was placed in service in August 1954. The roll itself was fabricated from three 2-in. thick curved Monel plates, weighing a total of 23,500 lb., which were welded longitudinally and then drilled with thousands of holes.

'After 2½ years of continuous service the Monel roll was in excellent condition and no shut-downs had been required for reaming or surface re-finishing. In addition, the fact that the surface of the roll remained smooth resulted in longer life of the felt.

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(2) For many years practically the only use for *tricresyl phosphate* was as a plasticizer for various resins, but its application as an additive in petrol has led to much increased demand. T.C.P. is usually made by reaction of cresol or cresylic acid with phosphorus trichloride, and nickel, which has been used for many years in the processing of phosphorus trichloride, has been found suitable also for vessels employed in purification of T.C.P. Reports of tests in crude tricresyl-phosphate storage containers and in the liquid phase of a tricresyl-phosphate distillation column are quoted to demonstrate the suitability of the metal for plant handling this corrosive medium.

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(3) *Ammonium sulphate* is an important source of nitrogen in the fertilizer industry. In the course of recovering ammonia (for subsequent production of ammonium sulphate), one of the large steel companies in Pittsburgh experienced difficulty in the operation of a condenser handling ammonia on the shell side and a corrosive river water on the inside of the tubes. Tubes of aluminium, copper and nickel-chromium stainless steel all gave unacceptably short lives. A close study of the problem led to selection of Inconel (nickel-chromium-iron alloy) for trial, and service experience has been so satisfactory that the condenser has now been entirely re-tubed with this material.

Corrosion Problems in Nuclear Power Reactors

W. Z. FRIEND: 'Corrosion Problems in Nuclear Reactor Power Stations.'

Reprint from *Proc. Amer. Power Conference*, 1956, vol. 18; 16 pp.

Issued by INTERNATIONAL NICKEL CO., INC.

The paper is introduced by a short review of the particular conditions in nuclear plant which complicate the selection of materials of construction. A brief summary is then made of some of the information which has been obtained by research and by experience in conditions relevant to those obtaining in reactors.

The report consists essentially of a review of the corrosion-resisting properties of groups of materials which are being used, or may be potentially useful, in nuclear-energy plant. The types considered are shown below, with some notes on outstanding characteristics as affecting their value in that field.

Austenitic Stainless Steels. These steels are already being used extensively and there is considerable evidence of their suitability. Some typical corrosion data, on four types of austenitic steel, in high-purity water at 500°–680°F. (260°–360°C.), demonstrate good resistance to attack. Warning is given of the risk of crevice corrosion and of stress-corrosion cracking of stainless steels in high-temperature water service, and recommendations are made for prevention of such deterioration. The sphere of usefulness of the precipitation-hardening austenitic steels, and their limitations, are also considered.

High-Chromium Stainless Steels. These steels are more susceptible to corrosion in conditions prevailing in nuclear water systems, and would have only limited application. Typical corrosion data are given in the paper.

Nickel and High-Nickel Alloys. Nickel and nickel-base alloys such as Inconel, Monel, Hastelloy B and C, and Illium are more subject to attack in oxygen-containing water at 500°F. (260°C.) than are the austenitic steels, but in de-gassed water, and particularly in hydrogenated water, they show very good resistance. Typical corrosion data, taken from reports of tests carried out under the aegis of the U.S. Atomic Energy Commission, are quoted for nickel, Inconel, Inconel X, Monel, K Monel, Hastelloys A, B, C and D, Illium, Incoloy and Invar.

Copper and High-Copper Alloys. These materials generally show heavy corrosion in high-temperature water containing oxygen. Under hydrogenated conditions their resistance is considerably better. Some data are presented for 70–30 cupro-nickel,

which has been much used for tubes of boiler feed-water heaters and for condensers in steam-power plants.

Carbon and Low-Alloy Steels. A review of the information already available indicates that carbon steel and some of the low-alloy steels might be suitable for use in reactor construction with pressurized water systems, but with the strict proviso that due regard were paid to the many complex factors affecting their behaviour.

The final section of this paper deals briefly with the conditions prevailing in boiling-water reactors.

Nitrided Stainless Steel for High-Temperature Water Service

K. ANDERSON: 'Nitrided Stainless Steels for High-Temperature Water Service.'
U.S. Atomic Energy Commission, Argonne Nat. Laby. Report A.N.L. 5192, Mar., 1954 (recently declassified); 89 pp.

During early work on systems requiring operation of parts immersed in high-temperature oxygenated water, difficulty was experienced in finding structural materials which would have the required combination of wear- and corrosion-resistance, machinability and mechanical properties. Preliminary tests verified the assumption that case-hardening would extend the useful life of many materials working under heavy loads, and would prevent galling. Of the various case-hardening procedures investigated, nitriding gave the most promising results, and a more extensive study of this method was therefore undertaken, on a co-operative basis, by the Reactor Division of the Argonne Laboratory, Battelle Memorial Institute, the Babcock and Wilcox Company, and Westinghouse Atomic Power Division. This report gives the results obtained in the respective laboratories.

Investigation of the suitability of the various nitriding processes showed that the most satisfactory results could be obtained by gas-phase nitriding, as exemplified by the Malcomizing method. (Surface-hardening by salt-bath treatment gave cases too thin and brittle to be useful on parts lubricated only by hot water.)

Study of stainless steels of various types indicated that precipitation-hardening stainless steels are likely to give the best service in the conditions envisaged: of those tested (Armco 17-4 P.H., and 17-7 P.H., and Stainless W), Armco 17-4 P.H.* proved the most suitable. Correctly nitrided surfaces showed wear-resistance markedly superior to that obtained with untreated steels. The nature of the pre-nitriding treatment, the nitriding conditions, and the post-nitriding treatment all had an appreciable influence on the resistance of the treated steel to corrosion in high-temperature oxygenated water, and the degree of attack was affected also by the initial temperature of the water and its oxygen content.

The major drawback to the use of nitrided steels was found to be the danger of couple corrosion, particularly at relatively low temperatures, e.g., about 200°F. (93°C.). This risk can, however, be minimized by suitable design; e.g., by arranging that a constant flow of fresh, neutral, high-resistivity water can be forced between mating surfaces during operation.

* For typical compositions, see p. 134.

Stainless Steel in Dounreay Atomic-Energy Plant

'Dounreay Atomic Energy Establishment.'
Engineer, 1957, vol. 203, May 17 and 24, pp. 747-51, 784-8.
Engineering, 1957, vol. 183, May 17 and 24, pp. 610-13, 665-8.

A detailed and well illustrated description of the experimental and research establishment at Dounreay. *Inter alia*, information is given on the materials of construction which have been selected for various critical components: for example, blanket elements of U-238 or Th are canned in stainless steel, and stainless steel is used for the reactor vessel (which is illustrated) and for the heat-exchanger system. Attention is drawn to the very exacting requirements to which fabrication of such plant must be carried out, and in this connexion some account is given of inspection methods adopted for welded piping and other components.

See also—

'Linear Induction Pumps for Liquid Metals.'
Engineer, 1957, vol. 203, May 17, pp. 752-5.

Discussion of requirements for successful operation of linear induction pumps which have recently been under intensive development for use in the atomic-energy field. The advantages of electromagnetic pumps are urged, as meeting the needs of various branches of industry, and the general principles and design of such units are discussed. Among the types of pump described, reference is made to units handling sodium or sodium-potassium at temperatures of the order of 250°C. and above. In these pumps, which, under test by the Atomic Energy Authority, are giving long and satisfactory service, most of the tubes are of 18-8 chromium-nickel stainless steel, with one in a Nimonic alloy.

Nickel-containing Materials of Construction in Uranium-Oxide Plant

T. W. WETT and G. W. TOMKIN: 'Safe Production of Enriched Uranium Oxide.'
Chemical Processing (*U.S.A.*), 1957, vol. 20, Jan., pp. 82-6.

Description, with flow sheet, of the uranium-dioxide plant operated by Mallinckrodt Chemical Works, Hematite, Mo., U.S.A.

The plant is designed to produce UO_2 nuclear reactor fuels from UF_6. Operation is in two sections, the one producing high-concentrate material (approximately 90 per cent. U-235) for reactors in which weight and size are critical (e.g., aircraft, submarines or locomotives), the other handling low concentrates, up to 3 per cent. U-235.

The following information is given with regard

to materials of construction which have been used in this plant:

'Lines from the UF_6 cylinder to the hydrolysis tank are of Monel; holding tanks, low-temperature drying trays and other lines are of low-carbon nickel-chromium stainless steel; trays used for 1500°F. (815°C.) firing and high firing are of Inconel or molybdenum.'

Resistance to Corrosion by High-Temperature Waters

C. J. LANCASTER: 'Corrosion Properties of Various Materials in High-Temperature Waters.'

U.S. Naval Engineering Experiment Station, Annapolis, Report E.E.S. 040028*B*. 15 pp + figures. *P.B.* 121,503.

The scope of the report is indicated below:

(1) Static tests on stressed and unstressed molybdenum-containing 18-8 and on 25-20 chromium-nickel steels in distilled water containing 300 p.p.m. of sodium chloride. Temperature 500°F. (260°C.).

(2) Static tests of stressed and unstressed molybdenum-containing and straight 18-8 chromium-nickel steels, in boiler feedwater of varying composition.

(3) Dynamic loop tests, at 10 ft./sec., in water containing 1-3 ml./L. of oxygen. Temperature 500°F. (260°C.). The following materials were tested in this series of experiments: nine types of austenitic chromium-nickel steel, including three precipitation-hardening grades; 90-10 and 70-30 copper-nickel alloys; Monel; Inconel and Inconel X (nickel-chromium-iron-base alloys); a Stellite alloy (cobalt-chromium-base), Hastelloy C (nickel-molybdenum-chromium-iron-tungsten alloy).

(4) Dynamic corrosion tests of various pre-stressed alloys, at 10 ft./sec. in natural sea water. Temperature 550°F. (287°C.). The materials used were those tested in (3) above, and titanium also was included.

Resistance of Nickel-containing Aluminium Alloys to Distilled Water

R. L. DILLON, R. E. WILSON and V. H. TROUTNER: 'High-Temperature Aqueous Corrosion of Commercial Aluminum Alloys.'

General Electric Co., Report HW-37636, Jan. 11, 1956; 14 pp.

The development, at Argonne Naval Laboratories, of the 1 per cent. nickel-aluminium alloy (see abstract in *Nickel Bulletin*, 1956, vol. 29, No. 7, pp. 145-6) has greatly increased the interest in aluminium-base materials for elevated-temperature service in distilled water, but until recently no comprehensive survey of the behaviour of commercially available aluminium alloys has been made.

The present report partially fills this gap, by presenting preliminary corrosion data on the resistance of 50 commercial alloys exposed in distilled water at temperatures of 250°-350°C. The test periods varied up to six weeks. Some of the alloys showed reasonably low corrosion rates up to 350°C.: others were satisfactory at 300°C. It was observed that all the more resistant alloys contained significant amounts of copper, nickel and silicon, in varying combinations.

Stainless Steel 'Sandwich' Material for Aircraft

V. C. SETTERHOLM and E. W. KUENZI: 'Performance of Stainless-Steel Sandwich Construction at High Temperatures.'

Wright Air Development Center, Report 55-417, Sept., 1956; 32 pp. *P.B.* 121681.

In the search for materials suitable for use in aircraft and guided missiles, stainless-steel 'sandwich' has proved promising for structural parts. The sandwiches consist of a lightweight core of stainless-steel honeycomb, with stainless-steel facings. At the request of Wright Air Development Center, several industrial organizations in the U.S.A. have been investigating the development of this type of material, and the properties which can be obtained in it by various processing and heat-treatment methods. The characteristics which are required include high strength/weight ratio, resistance to heat and to corrosion, fatigue-resistance, and a smooth surface finish which will be conducive to good aerodynamic performance.

This report contains results of tests on sandwich material made by six firms. The panels had all been made by brazing thin sheets of cold-rolled stainless steel to each face of a thin slice of stainless-steel honeycomb core. The panels varied with regard to type of stainless steel used, thickness of the facings, shape and size of core cell, method of making the core material, the brazing alloy used and the method of brazing, the thickness of the foil, and the heat-treatments which had been used on the sandwich material after fabrication.

Three forms of test were applied to the specimens: edgewise compression and flexure tests on sandwich material, and shear tests of both the composite sandwich and the honeycomb core steel. The majority of the compression and flexure tests were made after the materials had been heated at 75°, 800°, 1000° and 1200°F. (24°, 427°, 538° and 648°C.) for 10 minutes: some supplementary tests were made on material which had been at temperature for 192 hours. Shear tests were carried out at room temperature. Test equipment and technique are illustrated.

Although many of the panels were corroded, and poorly bonded, results of edge-wise compression tests showed that specimens from panels weighing only 2·2 lb. per sq. ft. can sustain facing stresses as high as 230,000 p.s.i. (102·5 tons per sq. in.) at 75°F. (24°C.) and 57,500 p.s.i. (25·65 tons per sq. in.) at 1200°F. (648°C.).

Stainless Steels for Pressure Vessels

A. GRODNER: 'Stainless Steels for Welded Pressure Vessels.'

Welding Jnl., 1957, vol. 36, Apr., pp. 169S-76S.

Abstract of an 'interpretive report' for the Materials

Division of the Pressure Vessel Research Committee. The full report is issued as Bulletin No. 31 of the Welding Research Council series; see abstract in *Nickel Bulletin*, 1957, vol. 30, No. 5, pp. 80–1.

Stainless Steel in Railway Engineering

'Six-Axle Twin-Car Diesel Trains.'

Engineer, 1957, vol. 203, June 14, p. 926.

A brief description is given, with dimensional details and an illustration, of a twin-car Diesel train built by Linke-Hofmann-Busch, G.m.b.H., Salzgitter, Germany. This train, which has 186 seats and standing room for 140 passengers, is stated to be the first to use an integral stress-bearing shell construction of plain, non-corrugated nickel-chromium stainless-steel sheeting. The twin-car set comprises two identical motor coaches arranged for two-directional operation. All the stress-bearing members are made from nickel-chromium stainless steel.

Laboratory Tests for Evaluation of Resistance to Sea-Water Corrosion

F. L. LAQUE: 'Theoretical Studies and Laboratory Techniques in Sea - Water Corrosion - Testing Evaluation.'

Corrosion, 1957, vol. 13, May, pp. 303t–14t.

This paper is based on the author's extensive experience of corrosion testing, which has included direction of the Kure Beach and other marine experimental stations in the U.S.A.

The paper describes the essential features of the principal laboratory tests which are being used for evaluating resistance to corrosion in sea water. The advantages and disadvantages of each of the methods are critically considered, with notes on precautions which must be taken in planning the respective types of test and in interpreting the results obtained.

It is pointed out that although sodium-chloride solutions offer certain advantages for use in laboratory tests, behaviour of specimens in such media is not strictly comparable with that shown in natural sea water. The author gives the composition of a synthetic sea water which has been found to give satisfactory approximation, and records results of tests correlating the corrosion rates of copper-alloy specimens in natural and in synthetic sea waters. The use of salt-spray tests is also discussed, with comparative data relating to corrosion tests of cold-rolled steel and zinc exposed to spray of natural and synthetic sea waters.

Apparatus used for simple-immersion tests is described and illustrated, and, in connexion with such tests, the influence of high velocity, turbulence and other variables is considered, with notes on specialized techniques such as jet-impingement. Other types of corrosion testing, to determine susceptibility to crevice attack, to galvanic corrosion, and to cavitation-erosion, are also described.

Attention is directed to the effect of salt water in lowering the resistance of metallic materials to repeated stress, and apparatus used in corrosion-fatigue testing is illustrated. The final section of the review is concerned with criteria for evaluating the relative corrosiveness of various waters.

The paper is supported by a selected *bibliography*, and an appendix contains the A.S.T.M. Standard Specification for Substitute Ocean Water (*A.S.T.M. Designation D*-1141-52).

Use of Electrochemical Methods in Metallographic Examination of Steels

C. EDELEANU: 'The Potentiostat as a Metallographic Tool.'

Jnl. Iron and Steel Inst., 1957, vol. 185, Apr., pp. 482–8.

A technique originally developed for the study of corrosion phenomena, in particular that of passivity in stainless steels, has been found to have useful application in metallographic study. The fundamental principles of the method were described in 1954: see *Metallurgia*, vol. 50, pp. 113–16: its use in metallography is the subject of the present paper.

It is demonstrated that the potentiostat, which can maintain the potential of a working electrode at any desired value, may be utilized in controlled etching, in detecting susceptibility to intercrystalline corrosion, and in the study of phase diagrams. Such applications are illustrated by reference to experimental examination of various types of austenitic chromium-nickel steel.

Copper-Nickel Casting Alloy for Marine Use

See abstract on p. 118.

S.G. Iron in Tankers

See abstract on p. 121.

Surface Treatments for Use on Low-Alloy Steels

See abstract on p. 112.

Properties of Thin Films of Nickel-Chromium Alloy

See abstract on p. 118.

PATENTS

Extraction of Nickel by the Carbonyl Process

Roasted nickel matte is subjected to fluid-bed reduction by pure hydrogen diluted with an inert gas, preferably nitrogen. The finely-divided flash-roasted sulphides are preferably diluted with sand

before reduction. The nickel carbonyl is then formed by reaction with carbon monoxide.

MOND NICKEL CO., LTD. (inventors, DE W. H. WEST and A. B. SIMPSON). *Brit. Pat.* 769,099.

Nickel-Activated Catalysts

Ethylene is polymerized in the presence of an aluminium trialkyl activated by nickel, cobalt or platinum. Nickel acetylacetonate is cited as one of the activating media.

K. ZIEGLER.
Brit. Pat. 773,536.
(similar to *Belgian Pat.* 527,736).

Nickel Interlay in Cladding with Zirconium

In cladding of steel with zirconium, electrodeposited iron or nickel is used as the interlay. Formation of a composite 'pack', for subsequent rolling, is described and illustrated.

LUKINS STEEL CO. *Brit. Pat.* 772,859.

Nickel/Boron-Nitride Composite

Shaped articles suitable for use as bearings and for other purposes are made from a mixture of a non-reactive metal (e.g., nickel, stainless steel, copper, tin, cobalt, or aluminium, used singly or in conjunction with one another) and boron nitride (1–5 per cent.). Examples cited include bars produced by hot-pressing a mixture of 95 per cent. nickel powder and 5 per cent. of boron nitride. (The term 'non-reactive metal' signifies a metal which does not react appreciably with or decompose boron nitride under the processing conditions involved).

CARBORUNDUM CO. *Brit. Pat.* 775,516.

Hard Metal/Carbide Products

A mixture of hard-metal carbides consisting predominantly of titanium carbide is recrystallized by heating and comminuting. The product is mixed with nickel powder and one or more other powdered binders selected in a proportion such that when the mixed mass is heated the binder metals alloy with one another and form a liquid binder-carbide eutectic with part only of the carbides. The balance of the carbides remains as a solid. The mixture is then comminuted, brazed and sintered. Typical examples of binders are given as chromium 50, nickel 50, per cent.; manganese 35, chromium 42·5, nickel 22·5, per cent.; iron-titanium (35–65,) 25, chromium 37·5, nickel 37·5, per cent.

L. NUSSBAUM. *Brit. Pat.* 774,817.

Activation of Nickel Electrodes for Alkaline Accumulators

According to the process claimed, the electrochemically active electrode material is thoroughly moistened with a cobalt-sulphate solution after having been precipitated in the form of a hydroxide, with or without a conductive additive. The material is then treated with caustic-soda, caustic-potash or an alkali- carbonate solution, and afterwards washed out with water. This process results in the formation of a water-insoluble cobalt compound which constitutes an effective barrier layer between the nickel-hydroxide particles.

VEB GRUBENLAMPENWERKE (inventor, H. WINKLER). *Brit. Pat.* 777,417.

Bright Nickel Plating

The brightening agent for an aqueous acidic nickel-containing electrolyte is an alkoxy coumarin containing an ethyl or methyl group. The basis solution is preferably of the Watts type. Typical compositions of modified baths are given.

UDYLITE RESEARCH CORPN.
Brit. Pat. 774,614 (similar to *Italian Pat.* 538,814).

Electroless Nickel Plating

In deposition by chemical plating from a solution containing nickel cations and hypophosphite anions, a light inert gas (preferably hydrogen) is dispersed in the bath to produce bright smooth nickel coatings on steel.

GENERAL AMERICAN TRANSPORTATION CORPN.
Brit. Pat. 777, 371.
Similar to *Austrian Pat.* 190,766.
Belgian Pat. 534,373.
French Pat. 1,116,849.
Italian Pat. 535,182.

Nickel Coating of Metals

Metals are coated by casting in a sand mould lined with a powder containing the coating metal and a flux. The lining of the sand mould preferably includes a resin bond with a metal-powder suspension. In an example given, a shell mould was sprayed with a suspension of nickel powder in an aqueous solution of barium chloride. The mould was dried and a steel bar was cast in it, thus being coated with nickel.

DIFFUSION ALLOYS, LTD. (inventor, R. L. SAMUEL).
Brit. Pat. 776,865.

Production of Oxalate Coatings on Nickel-containing Materials

Oxalate coatings are produced on materials containing high percentages of nickel and chromium by treatment with an aqueous solution of oxalic acid containing also a halide and/or a thiocyanate, and using an organic nitro-compound as accelerator. An example cited refers to the treatment of an 18–8 stainless steel in a solution containing oxalic acid 20, sodium thiocyanate 15, picric acid 2, per cent. Immersion was for 5 minutes, in a solution used at 170°F. (77°C.).

PYRENE CO., LTD. *Brit. Pat.* 769,779.

Nickel-Alloy Cathodes for Thermionic Valves

The detrimental effect of silicon in the nickel core of cathodes is eliminated by using a core of a nickel-manganese alloy (manganese 0·15-1·0, preferably 0·2-0·3, per cent.). The core is covered with a barium-containing alkaline-earth-oxide coating. Limits for impurities in the nickel-manganese alloy are given below:

	%
Magnesium	0·01—0·10
Silicon	0·05—0·10
Carbon	< 0·08
Sulphur	< 0·005
Copper	< 0·1
Cobalt	< 1·0
Iron	< 0·2

The nature of the detrimental influence of silicon and the mechanism of its correction by the method claimed are explained.

MULLARD RADIO VALVE CO., LTD. (inventors, W. E. F. COVENEY and C. H. R. GENTRY). *Brit. Pat.* 777,499.

Nickel-Silicon-Manganese Catalysts

Catalysts for hydrogenating rosin and rosin compounds consist of an alloy containing nickel 45-73, silicon 25-50, manganese 0·5-10, per cent. The particle size of the catalyst is specified as 0·062-0·05 in. in the maximum dimension.

The mass is activated by treatment with aqueous sodium hydroxide.

J. DREW and J. C. FUNDERBURK, assignors to HERCULES POWDER CO. *U.S. Pat* 2,739,947.

Tarnish Inhibitors in Cleansing Solutions

Tarnishing liable to occur on metals, particularly nickel-copper alloys and nickel silver, when in contact with soapless detergent solutions, is prevented by incorporating in the cleanser certain acyl dicyandiamides. A preferred addition is lauroyl dicyandiamide.

UNILEVER, LTD. (inventor, A. D. SCOTT). *Brit. Pat.* 772,493.

Magnetostrictive Alloys

Improved magnetostrictive elements for use in converting electrical to mechanical energy contain cobalt 15-20·5 per cent., nickel balance. Such materials may be used as transducers in echo-sounders and in ultrasonic drilling apparatus.

MOND NICKEL CO., LTD. (inventor, C. A. CLARK). *Brit. Pat.* 774,727.

Magnetostrictive Alloys for Use in Filters

In electromagnetic filters, as described in British Patent 722,664, wires are used for coupling energy to various parts of the filter. The magnetostrictive alloy used should possess a low temperature coefficient of frequency, high internal friction, and high magnetostrictive-frequency response. These characteristics are obtained in an alloy containing nickel 44·2, iron 55·7, manganese 0·1, per cent., melted in an inert atmosphere, cast, swaged, drawn to wire and dead-soft annealed in dry hydrogen, preferably at 2000°F. (1093°C.).

COLLINS RADIO CO. *Brit. Pat.* 772,564 (similar to *U.S. Pat.* 2,719,084).

Nickel-Alloy Steel for Sub-Zero Service

The patent covers a relatively inexpensive steel which requires only a simple heat-treatment and is capable of giving, at sub-zero temperatures, impact values above 15 ft.-lb. in Charpy keyhole tests. The composition limits are given as carbon 0·02-0·15, silicon 0·05-0·6, manganese 0·2-1·2, copper 0·2-1·2, chromium 0·25-1·5, nickel 1-5, aluminium 0·03-0·3, nitrogen 0·01-0·025, tantalum 0·04-0·25, per cent., iron balance. A small percentage of molybdenum may also be present.

UNION CARBIDE LTD., assignee of M. KORCZYNSKY. *Brit. Pat.* 769,871.

Heat-Treatment of Nimonic Alloys

A known heat-treatment for alloys of the Nimonic type comprises heating for 1-12 hours at 1050°-1200°C., cooling in air or quenching in oil or water, and ageing by re-heating for 12-18 hours at 650°-750°C., followed by air-cooling. According to the claim made in this patent, an additional heat-treatment, carried out after the above cycle, confers substantial improvement in tensile strength and fatigue-resistance of the alloys at temperatures above 700°C.

The further steps, which form the subject matter of the patent, consist in heating the alloys at 800°-1000°C. for a period up to 10 hours, cooling in air or quenching in oil or water, and subsequently heating at 600°-900°C. for a period up to 10 hours. An example of the total heat-treatment cycle is cited as follows:—Heating for 6-10 hours at 1050°-1100°C., cooling in air, ageing at 690°-710°C. for 12-18 hours, cooling in air, and then subjecting the alloys to further treatment consisting of heating at 930°-970°C. for 3-10 minutes, cooling in air, and finally heating at 780°-820°C. for 1-2 hours. Typical properties are given for alloys treated by these modified cycles.

BRISTOL AERO-ENGINES, LTD. (inventor, P. H. FRITH). *Brit. Pat.* 777,703.

Boron-containing High-Temperature Alloys

Alloys claimed to be resistant to temperatures of the order of 900°C. contain cobalt 30-45, nickel 20-35, chromium 15-20, tungsten 1-7, molybdenum up to 5, tantalum up to 5, niobium up to 3, titanium 3-9, carbon 0·1-2, boron 0·005-0·6, per cent. Preferred compositions are given and the tensile, fatigue and creep properties of the materials are

discussed. Homogenization treatment at 1180°C. and hardening at 850°–900°C. are also claimed.

CIE. FRANCAISE THOMSON-HOUSTON (inventor, P. J. CLEMM). *French Pat.* 1,131,776.

Complex High-Carbon Nickel-Chromium-base Alloys for High-Temperature Service

Complex nickel-chromium alloys suitable for service under stress at 600°–1000°C. are characterized by specially high carbon contents. Limits of composition are given as follows: carbon 0·5–2·0, manganese 0·1–2, silicon 0·1–2, chromium 15–25, tungsten 1–5, molybdenum 1–5, niobium and/or tantalum 1–6, iron 0–15, per cent., nickel balance. Boron up to 0·5 per cent. may be present and the alloy may also incorporate the following elements, used in amounts up to 3, per cent. singly or in combination: aluminium, beryllium, cerium, copper, titanium, vanadium and zirconium. Nitrogen 0·01–0·2 per cent. may also be present.

W. JESSOP AND SONS, LTD. (inventors, G. T. HARRIS and H. C. CHILD).

Brit. Pat. 773,871 (similar to *Australian Pat.* 203,523; *Belgian Pat.* 532,001; *French Pat.* 1,106,645).

High-Temperature Alloys Resistant to Thermal Shock

Alloys highly resistant to thermal shock contain carbon 0·2–0·85, cobalt 5–15, chromium 18–25, molybdenum 8·5–12, per cent., nickel balance. Impurities for which individual limits are given are aluminium, titanium, iron, silicon and manganese; it is further required that the total of these impurities shall not exceed 2 per cent. Thermal treatment is also covered, comprising solution treatment at 1200°–1280°C. for 1–4 hours, followed by air cooling.

ROLLS-ROYCE, LTD. (inventors, H. E. GRESHAM, M. A. WHEELER and J. R. BIRD).
Brit. Pat. 770,909.

Heat-Resisting Steels Immune to Over-Ageing

Austenitic steels suitable for regular use at temperatures of at least 650°C., and insensitive to overheating, are characterized by a titanium content which bears a definite relation (*a*) to the sum of the iron and cobalt contents and (*b*) to the content of carbon+nitrogen. The broad compositional limits claimed are nickel and/or manganese 8–65, iron (with or without cobalt) at least 20, carbon up to 0·6, chromium 13·7–30, silicon 0–2, per cent., and titanium in such amount that the 'active titanium' is not more than 2 per cent. The possible presence of nominal amounts of other elements is specified, and typical compositions are cited. Hot working at a temperature preferably above 1000°C., with subsequent re-heating to about 1100°C., is claimed to improve the heat-resisting properties.

(*Note.*—'Active titanium' is defined as the titanium which is not combined with carbon and nitrogen.)

NYBY BRUKS AKTIEBOLAG (inventor, A. R. WAGNER). *Brit. Pat.* 777, 034.

Nickel-Alloy/Precious-Metal Thermocouple

In a bimetallic element, the high-expansion member is made from a nickel-chromium alloy (e.g., 80–20 type) which may also contain small amounts of other elements, and the low-expansion member is a platinum-base alloy containing 20 per cent. of rhodium.

BAKER AND CO., INC. (inventors, M. J. STUMBOCK and W. L. PERRINE).
Brit. Pat. 730,385.

Thermocouple Elements Resistant to Furnace Atmospheres

Thermocouples covered by the patent consist of an electropositive element made from a nickel-chromium-base alloy containing additions designed to improve its resistance to high-temperature corrosion and a negative element made from a nickel-base alloy containing manganese, aluminium, silicon and cobalt in small proportions. Typical compositions are given as follows:

Electropositive element: chromium 19·5, carbon 0·06, silicon 0·8, manganese 0·06, niobium 1·1, per cent., balance essentially nickel.

Electronegative element: manganese 2·90, carbon 0·01, silicon 0·99, aluminium 1·99, per cent., balance essentially nickel.

Characteristics of thermocouples of this type are discussed.

BRITISH DRIVER-HARRIS CO., LTD.
Brit. Pat. 773,919.

Production of Stainless-Steel Powder Compacts

Finely divided nickel powder (preferably of particle size not greater than 53μ) mixed with a lubricant (preferably paraffin wax and/or calcium stearate) is added to stainless-steel powder prior to sintering.

BIRMINGHAM SMALL ARMS CO., LTD.
French Pat. 1,136,174.

Welding of Austenitic Nickel-Chromium Steel Tubing

Tubes made from fully austenitic niobium-containing nickel-chromium steel are butt-welded by depositing on the ends partially ferritic niobium-containing nickel-chromium steel, and maintaining a super-atmospheric pressure inside the tubes while welding the built-up ends in an inert-gas atmosphere. The deposits are preferably of 9-19-Nb nickel-chromium steel on 8-18-Nb steel.

BABCOCK AND WILCOX, LTD. *Brit. Pat.* 769,256.

Austenitic/Ferritic Welded Joints

In welded assemblies consisting of an austenitic steel member and a ferritic steel member, designed for operation at high temperatures, carbon migration from the ferritic steel is prevented by the use of a carbide stabilized ferritic alloy steel weld deposit and a heat- and corrosion-resisting overlay. The first, 'buttering', layer may be, for example, a low-

alloy chromium-molybdenum steel, and Inconel may be used for the overlay.

BABCOCK and WILCOX, LTD. *Brit. Pat.* 774,967.

Brazing of Molybdenum and Molybdenum Alloys

Temperatures involved in the welding of molybdenum and molybdenum alloys are liable to produce embrittlement. This effect is obviated by brazing with a metal which is capable of alloying with molybdenum, e.g., nickel. To confer oxidation-resistance on the joints so made, a siliciding treatment is applied.

BRITISH THOMSON-HOUSTON COMPANY, LTD. (inventors, W. J. SCOTT, H. RAWSON and H. J. CHAFER). *Brit. Pat.* 774,291.

Attention is drawn to the fact that many of the names of materials mentioned in The Nickel Bulletin are *Registered Trade Marks*.

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